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Complete Recycling of Textile Components From Cotton/Polyester Blended Fabrics.

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COMPLETE RECYCLING OF TEXTILE COMPONENTS FROM COTTON/POLYESTER BLENDED FABRICS

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LIST OF ABBREVIATIONS

ACER: advanced capillary extrusion rheometer

CT: cotton

DP: degree of polymerization

DMS: dynamic mechanical spectrometer

DMSO: dimethyl sulfoxide

DSC: differential scanning calorimeter

DTA: differential thermal analysis

N: nylon 6,6

NaOCl: sodium hypochlorite

NaOH: sodium hydroxide

NMMO: N-methyl morpholine N-oxide

NMMO·H_2O: N-methyl morpholine N-oxide monohydrate

PET: polyester (polyethylene terephthalate)

R: rayon

ABSTRACT

The purpose of the study was to investigate the possibility of recycling textile components (especially cellulose) from cotton and polyester blended fabrics. The separation methods of intimately blended fibers developed were: 1) hydrolysis of polyester in NaOH solution or 2) dissolution of cotton in an N-methylmorpholine N-oxide monohydrate system. Complete separation was confirmed with thermal analytical methods such as differential scanning calorimetry and thermogravimetric differential thermal analysis. These thermal methods were found to efficiently determine accurate fiber contents in cotton/polyester blends and in rayon/nylon 6,6 composite fibers. In the recycling process for cotton/polyester blends, a 4M NaOH solution at boiling temperature and 2% NaOCl for an hour were optimum conditions for hydrolysis of polyester and bleaching of cotton residues respectively. Rheological characterization of 15% and 17% recovered cellulose solutions in the solvent system was carried out in a dynamic mechanical spectrometer which provided important information on conditions for spinning of high tenacity fibers. Lyocell fibers from the recovered cellulose solution were spun using an advanced capillary extrusion rheometer, and then dyed with a direct dye and a reactive dye.

1. INTRODUCTION

The awareness of environmental problems has steadily increased during the last two decades. Environmental protection activities started from recognition of the close relationship between human life and the natural environment. Individuals' perception of risk caused by environmental problems has resulted in a variety of environmental protection activities. Material recycling, including textile materials, can be regarded as one of the active environmental protection activities due to the economic and ecological effects of newly developed material recycling technologies.

A great deal of effort has been made to recover and recycle raw textile fiber and post-consumer textile materials. The category "textile" usually includes old clothing, bedding materials, towels, furniture cushions, carpets, nylon shower curtains, and rags. Over four million tons of post-consumer textiles enter the waste stream annually (5.3% of total municipal solid waste during 1990). A quarter of the annual post-consumer textile waste stream has been collected for recycling, and half a million collected textiles were sold and reused for the same function for which they were originally produced [30]. For example, used clothing has been generally wholesaled or retailed by the pound or piece through charitable organizations, rag industries, or recyclers.

The serious environmental problems of most solid waste materials are overloaded landfills, air pollution from incineration, and destruction of natural

ecosystems. Textile and apparel manufacturers are increasingly involved with research and development of recycling technologies, and producing a variety of marketable recycled products. Other environmental activities in the textile and apparel complex are minimization of fiber waste in yarn manufacturing processes and sewing and cutting waste in apparel manufacturing because raw material and waste disposal fees are increasing. The re-use of post-consumer soda bottles to produce new textile materials is one of the best examples of recycling.

As most apparel on the market today is of mixed fiber content, the most significant problem of efficient textile recycling is lack of an economical separation method for post-consumer textile materials according to fiber content. The most common blend in the apparel market is cotton(CT)/polyester(PET: polyethylene terephthalate). In this research, separation techniques for CT/PET blended textile materials were investigated to develop processes that maintain the inherent physical and chemical properties of CT and/or PET fibers. Subsequently, specific optimum conditions and procedures were determined for complete recycling of the CT and PET components in blended fabrics. As much research has been done to recycle PET, the focus of the second part of this study was on recycling the separated CT component from CT/PET blends. A recycling procedure of the separated CT residues was investigated to produce a lyocell fiber by using an environmentally safe solvent system.

1.1. Objectives

1. To develop methods for separating CT and PET fibers in blended fabrics.

2. To determine the efficiency of the developed separation methods.

3. To extrude a new lyocell fiber from the cellulose separated from CT/PET blends.

3-1. To determine the characteristics of the recovered CT solutions for spinning.

3-2. To bleach the CT residue and the extruded fibers from recovered CT.

3-3. To dye the extruded fibers from recovered CT.

1.2. Limitations

1. The current study was limited to the investigation of recycling of CT/PET blends only.

2. Since the technology for recycling pure PET is already commercial (for example, recycling of PET soft drink bottles), the present investigation was limited to degradation of PET into starting polycondensation monomers, i.e., terephtalic acid (solid crystalline material) and ethylene glycol (acidic aqueous solution).

3. Recycling of CT was limited to solvent spun lyocell-type fibers prepared from recovered CT.

2. REVIEW OF LITERATURE

2.1. Cotton and polyester

2.1.1. Cotton

CT is the world's most widely used fiber. Its popularity stems from its relative ease of production and its applicability to a wide variety of textile products. CT fibers range from 0.3 cm to 5.5 cm in length, and appear microscopically as ribbonlike structures with irregular convolutions and a kidney-shaped cross-section. The CT fiber is composed of high molecular weight cellulose in a well-ordered structure of linear chains. The basic repeating unit of cellulose is cellobiose, which is two beta-glucose monomer units linked by an oxygen atom. CT fibers are composed of highly hydrogen-bonded elementary fibrils and random nonparallel polymeric aggregates. The neatly packed crystalline regions lead to high strength, low elongation and low pliability along with high orientation, while the amorphous regions are responsible for the diffusion of chemical finishes or dyes [6]. The hydroxyl groups of cellulose are the chemically reactive unit in application of dyestuffs and finishes.

CT's softness and absorbency translate into comfortable textile products. The fiber is decomposed by acid solutions, especially strong mineral acids; however, it has excellent resistance to alkaline solutions. It launders readily and can withstand strong detergents, high temperatures, and bleaches. It is seldom damaged by insects, but may be destroyed by fungi, such as mildew.

Fabrics of many different constructions, weights, and textures can be made from CT fibers. Examples include apparel fabrics, household or domestic goods, home furnishings, and industrial applications. Synthetic manufactured fibers are often blended with natural or manufactured fibers to enhance their dyeability, comfort, and absorbency and to reduce static charge. An important environmental advantage of CT is its biodegradability.

2.1.2. Polyester

Polyester, introduced in 1953, is now the most used manufactured fiber in the world. To meet the demand of textured yarn and fabric manufacturers, many variants of polyester have been developed with distinct characteristics such as crimp, strength, dyeing characteristics, softening and melting temperatures, cross-sectional shape, whiteness, or other properties that enhance yarn processing or behavior of end-use products.

The Textile Fiber Products Identification Act (TFPIA) defines polyester as: a manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 85% by weight of a substituted aromatic carboxylic acid, including but not restricted to substituted terephthalate units and parasubstituted hydroxybenzoate units [34]. The three most important polyester fibers are PET, poly 1,4-cyclohexane dimethylterephthalate (PCDT), and polyethylene oxybenzoate (PEB) [57]. The most widely used polyester fiber is PET. It is produced by a stepwise polycondensation reaction between ethylene glycol and terephthalic acid. Commercial production is by transesterification and polycondensation of dimethylterephthalate with ethylene

glycol [11]. PET is free of toxic combustion products other than carbon monoxide [29].

The fiber is produced by melt spinning in staple or filament form. Polyester fibers are smooth, solid rods with round cross-sections. The properties of polyesters are determined by hydrophobic aromatic rings and ester groups in the molecule, resulting in low moisture absorbency and generation of static charge. The tenacity of polyester is in the range of 2.5-9.0 g/den, and its density is 1.38 g/cm^3. Polyesters have many desirable performance properties such as strength, dimensional stability, and wrinkle recovery. Good chemical properties include a strong resistance to organic solvents and all common acids and alkalis except hot sulfuric acid. The glass transition temperature and the melting point of polyester are in the range of 175-200°C and 238-290°C, respectively.

Polyester fabrics have been widely used in apparel, home furnishings, and various commercial and industrial applications. Polyester is the most favored manufactured fiber to blend with CT, wool, rayon or flax fibers for many different end-use products because of advantages such as easy maintenance, strength and durability, abrasion resistance, wrinkle-free appearance, and shape and size retention.

2.1.3. Cotton and polyester blends

The introduction of polyester and the shrinking availability of CT growing acreage around the world resulted in CT's declining importance in the world markets in the 1950's through the 1970's [34]. However, increasing public

environmental awareness about ecological effects of non-biodegradable materials has brought back the use of natural fibers, especially CT. Shirts, sheets, underwear, dresses, pants and other apparel which were once 100% CT are now CT/polyester blends to compensate for the poor wrinkle resistance of CT. For instance, 65% polyester/35% CT fabrics give improved comfort and better dyeability as well as wrinkle resistance. In general, CT contributes good moisture absorption and hand, while polyester improves fabric performance properties such as wrinkle resistance, elasticity, dimensional stability, strength, abrasion resistance, and shorter drying times.

2.2. Recycling of textile materials

2.2.1. Post-consumer textile recycling

Recycling is the recovery for reuse of the economic values of materials and energy from wastes that are usually destined for disposal [2]. Textile materials are usually components of municipal solid waste. In 1990 over four million tons of post-consumer textile materials entered the waste stream annually [30]. Only a quarter of the annual post-consumer textile waste is collected for recycling and half a million collected textiles were sold through charitable organization stores, the rag industry or recyclers. One recent study indicated that an average of about 250,000 pounds of post-consumer textile materials were received each month by local charitable organizations which in turn sold the largest amount of donated clothing to the rag industry [20]. The other disposition method of donated clothing is sale through thrift store operation, with only a small portion sold to recyclers.

It has been known that most textiles collected in the US are usually reused for the same function as the virgin products. For example, used clothing is generally wholesaled or retailed by the pound or piece to the general public in the developing countries. There are approximately 200 companies at the primary processing levels and an additional 150 secondary processors such as used clothing exporters, wiper manufacturers, and fiber and fabric manufacturers active in recycling post-consumer textile waste [47].

The majority of used textiles collected in the US utilize a drop-off box or telephone routing system. A pilot study examined the inclusion of textile materials in existing curbside collection programs and identified problems such as the low rate of generation and recovery of textile materials for new materials and the necessity of an additional in-house sort and set-out [30]. In addition, there are some other specific collection problems: collection personnel's trouble in differentiating bagged textiles from regular bagged waste, additional care of keeping textile waste dry, and early distribution of relatively contaminant free plastic bags. In spite of those troubles, the current active textile recycling has contributed to environmental preservation by introducing new products for alternative uses. Examples are nonwovens in automotive, furnishing, carpet, agricultural and other industries [58].

2.2.2. Textile recycling in textile/apparel complex

Textile and apparel manufacturers and retailers are very closely related businesses as they influence one another and are influenced by consumers. Therefore, the entire textile industry complex has accepted recycling activity

and promoted high quality recycled products. Textile manufacturers identified their recycled products by favorable names such as "eco," "green," or "environmentally friendly" to alert consumers to their environmental interest and successful technologies [1,48,63].

The primary concern of the apparel manufacturing segment has been cutting waste according to the American Apparel Manufacturers Association's Technical Advisory Committee in a 1993 survey [62]. Cutting waste, the waste fabrics from cutting of garment pieces, reached 176 million pounds annually, the equivalent of nearly seven yarn mills (at a production of 500,000 pounds yearly) running all year, and 166,300 acres of CT production (at an average of 635 pounds/acre). Another practical comparison was that a major knit producer generated an average of 21.5 million pounds per year of waste from cutting and sewing processes.

Problems encountered by textile manufacturers in material recycling include the difficulty of producing new products, lack of processing equipment, and the cost of recycled products in the market [25]. With those economic barriers of recycling textile fibers, textile recycling is not cost effective at this time. Many textile and apparel manufacturers, however, have been working cooperatively to minimize waste and to improve recycling of materials [12]. Wellman Inc., a pioneer in making PET fiber from discarded PET soda bottles, makes textile fibers for carpets and fiberfill. The company has also developed a finer denier fiber for Ecospun Fortrel. DuPont is developing a program that will make virgin-like PET out of post-consumer PET products [38]. Hoechst

Celanese has introduced Trevira II, a blend of 50% post-consumer plastic bottles and 50% virgin PET [49].

The possibility of recycling CT yarn was introduced in manufacturing high quality yarns by blending with virgin CT fiber or low quality recycled yarns [28]. Burlington industries has made 50% virgin CT and 50% reclaimed denim fabrics, which are fashionable and environmentally friendly. Dixie Yarns offers recycled yarn made from waste fibers in the spinning process [49]. Wellington Sears recycles byproducts from virtually every textile process from carding through weaving and cutting [44]. Its recycled products are used for mattress insulation, furniture stuffing, cushioning, industrial wiping cloths, and sound deadening in automobiles [65]. Malden Mills, an early manufacturer of recycled textiles, is currently selling about 6 million yds of recycled yarn fabric to the outdoor market [48]. Malden uses recycled fiber developed by Hoechst Celanese, Wellman and DuPont.

Dyersberg Fabrics and Patagonia started working with recycled fabrics in order to make an environmental statement to the outerwear market [48]. These recycled textile products proved good enough to satisfy the high standards for quality and performance as well as the customer demand for environmental assessment. Swift Textiles has become famous for its community and customer promotion program around its "Soda Pop Denim" made of 80% CT and 20% PET. This company has formed a partnership to emphasize the health and economic benefits of recycling with Lee Apparel, a school district, and a department store to promote the product.

Apparel manufacturers and retailers are adding impetus by adopting corporate environmental policies. Companies such as L.L. Bean, Levi Strauss, Wrangler, The Gap, Patagonia, K-mart, JC Penney and Wal-Mart visit suppliers' facilities to review wastewater treatment operations and recycling programs [44].

2.2.3. Supports for recycling activities

The environmental Protection Agency (EPA), the initial driving force of all environmental protection activities in the textile industry, has implemented a special program to increase the growth of recycling by linking economic and environmental priorities. The Jobs Through Recycling Initiative is a $2.7 million grant program designed to increase the use of collected materials by creating markets for recycling industries, stimulating economic development and fostering the creation of new jobs [19]. Another specific textile recycling effort is the American Textile Manufacturers Institute's Encouraging Environmental Excellence (E^3) program. This is one example of initiating recycling and waste minimization activities in major textile corporations and trade organizations [62].

2.3. Fiber production technologies for cellulosic materials

2.3.1. Viscose rayon process

The earliest, and the most common commercially regenerated cellulosic manufactured fiber was produced by the viscose process. Viscose rayon has become one of the most important manufactured fibers and has been used extensively for blending with other natural or synthetic fibers, or for making

lining fabrics or fashionable crepes. End-uses for viscose rayon expanded gradually, only to be eclipsed when the first truly synthetics, such as nylons and polyesters, entered the market [59].

The process of making viscose rayon begins with formation of soda cellulose by steeping and pressing in 18% caustic soda solution [37]. The moist mass of cellulose is followed by shredding and aging for a half to one and a half days in order to reduce the degree of polymerization (DP) in cellulosic molecules and to insure complete conversion to soda cellulose. The shredded crumbs are formed into sodium cellulose xanthate by reaction with carbon disulfide (CS_2). The cellulose xanthate is then dissolved in aqueous sodium hydroxide (NaOH) solution to yield the viscose solution. When the viscose solution is extruded into a coagulation bath containing sulfuric acid (H_2SO_4), the regenerated cellulosic fiber is formed and then drawn to increase orientation and strength. This viscose rayon production is known to be a complex process that requires strict control of the byproducts formed at different stages. The hydrogen sulfide (H_2S) gas and H_2SO_4 waste effluent were always the major sources of pollution. As legislation for environmental protection imposes tighter restrictions, this viscose rayon process becomes more and more costly, limiting economic viability and reducing the number of producers.

2.3.2. Solvents for cellulosic materials

It is reported that the recently developed N-methylmorpholine N-oxide (NMMO) solvent spinning process of cellulose has been successful in producing new fibers with many favorable properties [15]. The concept of

spinning cellulose fibers from solvents was proposed over sixty years ago [24], but it was not until the late 1960s and early 1970s that patents began to be filed in this area [31,33]. By 1979 and the early 1980s the use of amine oxide was attracting attention from manufacturers who had begun to look for new routes for cellulosic fiber production giving improved fiber properties and performance [22,40,42]. Gradually researchers came to focus on the advantages of NMMO over other solvents.

Solvent systems are generally categorized into derivatizing and non-derivatizing systems. Materials that exhibit strong intermolecular hydrogen bonding, such as cellulosic and protein fibers, are insoluble in commonly used solvents except under the extreme conditions of high temperatures and strong acids or bases that lead to degradation of the compounds. The high molecular weight and crystallinity of the fibers, as well as hydrogen bonding and solubility parameters, are important factors to be considered in dissolving cellulose [64].

The new NMMO solvent system is a non-derivatizing system that dissolves the polymer by intermolecular interaction only by the formation of hydrogen bond complexes [46]. In theory, amine oxides succeed in effecting solution of cellulose because their steric characteristics allow the polar or oxide end of the solvent molecule to solvate the polar hydrogen bonds of the compound while the rest of the solvent molecule can solvate the organic or non-polar bonds [24]. Thus, complete solvation and consequent solution can take place.

The amine oxides are produced from a class of tertiary amines that are easily and efficiently oxidized to the corresponding N-oxides by reacting those amines with hydrogen peroxide in the presence of unstable inorganic per-compounds of acid-forming elements from groups VA, VIA, VIB and VIII of the periodic table as catalysts [66]. The resulting compounds such as NMMO, N-methylpiperidine-N-oxide, N-methyl-pyrrolidine-N-oxide and N-methylazacycloheptane-N-oxide were found to dissolve cellulose [50].

Because of the inert nature and neutral pH of the amine oxides and the low temperatures to effect solution, the dissolved compound is neither degraded nor chemically changed. This is reflected in the high viscosity of solutions of compounds dissolved in the amine oxides. After dissolution of cellulose in the NMMO solvent system, crystallization of NMMO and cellulose occurred subsequently forming a network of cellulose crystals on the solvent crystals [13]. This structure is responsible for the characteristic microfibrillar texture of the regenerated cellulose matrix. This cyclic amine morpholine compound does not yield color nor a potentially dangerous residue in the product [32,43].

The amine oxides are themselves soluble in most common solvents such as water, methanol, acetonitrile, toluene, xylene, dimethylformamide and dimethyl sulfoxide (DMSO) [41]. Thus, the amine oxides can easily be reclaimed from fibers or films made from solutions with various compounds. The addition of water or DMSO was effective in achieving a homogeneous solution by slowing down the dissolution so that amine oxide is absorbed

uniformly [40]. A cellulose solution in amine oxide was also adapted to be extruded or spun at higher temperatures [42].

A solvent containing the tertiary amine N-oxide formed a precursor solution of cellulose which was spun, extruded or molded at a temperature below the melting point of NMMO (73°C). A solution of cellulose with a relatively high solid content (10-25% by weight) in a tertiary amine oxide was suggested for shaping materials such as extruded parts or spun fibers [21]. Many modified fibers can be produced in this solvent spinning process with additives. When ammonium chloride (NH_4Cl) or calcium chloride ($CaCl_2$) were added, a substantial increase was made in the viscosity of the NMMO dissolved spinning cellulose [14]. A chip or similar granular cellulosic product was also made for convenient storage and formation of cellulose solution [22]. Because the solvent does not react with the cellulose, the solvent can be fairly easily recovered for recycling.

The recently developed solvent spinning process of cellulose has been successful in producing new fibers through a closed recycle-recovery loop spinning process [36]. In the solvent process, the spinning solution with crushed wood pulp is prepared, and then passed to a continuous dissolving unit to yield a clear, viscous solution. It is extruded into a dilute aqueous solution of amine oxide which precipitates the cellulose as a lyocell fiber. Because NMMO is an expensive solvent, the economics of the process dictate recovery and recycling. This is done by purifying the NMMO in ion exchange columns and removing excess water. The entire process utilizes materials which are

environmentally safe and, since the solvent is recycled, the waste products are both minimal and non-hazardous [15].

2.3.3. Lyocell fiber

There is still considerable appeal in the concept of making fibers from cellulose: the resource for the raw material is plentiful and renewable and the fiber resembles natural fibers more than do other manufactured fibers made from synthetic polymers. Therefore, work on other routes to cellulosic fibers has concentrated on a process involving direct dissolution of cellulose. An amine-oxide derivative, NMMO is one of the solvents identified to be safe in the environment in the event of any losses and almost completely recyclable during processing. Fibers spun from solutions of cellulose in NMMO have now been generically designated as lyocell fibers to distinguish them from rayon. Both British viscose producer, Courtaulds, and the Austrian company, Lenzing, set up pilot plants in the last decade with the objective of developing the lyocell spinning process from NMMO/cellulose solutions. By 1995 Courtaulds had completed a commercial scale plant in Axis, Alabama, producing about 50,000 tons/year, while Lenzing has decided to build a large production line in Heiligenkreuz, Austria, with start-up scheduled for mid-1997.

The Federal Trade Commission has proposed recently to allow clothing manufacturers to use the generic name, lyocell, in fiber content labels. This is because solvent spun lyocell fibers, although substantially the same as rayon in terms of chemical composition, have significantly different characteristics. Lyocell fiber is strong especially when wet; it is easy to spin into fine yarns, and

is stable in washing and drying while rayon is not machine-washable [67]. However, one possible concern that accompanies lyocell fibers is that of fibrillation under conditions of wet abrasion [15]. Under mechanical stress or when swollen with water, there is a tendency for the fiber to divide into fine fibrils, due not only to its high orientation, but also to its apparent lack of lateral cohesion. This adversely affects the launderability of the product in many textile end-uses, but in other markets (such as nonwovens) it proves to be an advantage, having a certain appeal for making hydroentangled materials. In fact fibrillation-resistant fibers can already be made, but these tend to result in textiles with a harsher hand than is desired.

Lyocell fibers are typically spun from a solution of cellulose in a mixed solvent of NMMO and 13-19% water (essentially NMMO monohydrate) between 85-100°C, a temperature domain much above the melting point of the monohydrates (73°C). An anisotropic liquid crystalline (LC) can be developed if the cellulose concentration is high enough (higher than 15%, depending on DP of the cellulose chains) [67]. Formation of anisotropic phases, which can result in higher tenacity fibers, should be flow-induced at lower concentrations, depending on the rheological conditions of fiber extrusion.

The developed Courtaulds' lyocell staple fiber, Tencel, has a linear density range of 1.1-3.3 dtex. The special properties of lyocell fibers include high dry/wet tenacity, good dyeability, and controlled fibrillation. Because of the relatively high current price, Tencel is blended with CT, linen, silk, or other fibers in making competitively marketable fabric products [49].

2.4. Recycling technologies for PET

Plastic has symbolized the most visible solid waste in our society. Since plastics require long periods of time to disintegrate and take up a large volume of space in landfills, finding ways to recycle them and manufacture biodegradable materials has become an important environmental priority and the key to inhibiting anti-plastic regulation.

PET is a clear, tough, non-toxic material used to make virtually all plastic soda bottles, containers for vegetable oil, polyester fabrics, and certain other goods. The recycling technology of PET soda bottles into nonwovens has been successful [61]. The markets for PET soda bottles and clear high density PET have been strong, and the recycling of those materials is also reported to be cost effective because of the value and quantity of recycled plastic [17,51].

Depolymerization is one technology for reuse of plastic materials. In the first step of this process, the collected plastic waste materials are converted into free flowing granules. In a continuous process the chips from widely different thermoplastic materials and their blends are remelted [23]. Some methods such as froth floatation and density separation have been used in separating plastic materials depending on the generic types. A device was also designed for the purpose of efficient separation of PET [8]. It allows low density foreign materials to float to the surface of the melt and high density foreign materials to sink to the bottom of the melt, and remove molten PET oligomer from an area which is located intermediately between the surface of the melt and the bottom of the melt.

The advantage in depolymerizing PET is that ester bonds in PETs can be rapidly hydrolyzed to ethylene glycol and a diester of terephthalic acid under relatively mild conditions with a mixture of alcohol (glycol), a polar solvent (N-methylpyrrolidone or DMSO), and an alkoxide or hydroxide [39]. One such an example was aqueous hydrolytic depolymerization of condensation polymer waste material at 200-300°C. Ammonium hydroxide in place of the stronger alkali metal bases, such as NaOH, was also used in a depolymerization process of PET [35]. The recovered component materials were then repolymerized to form new PET products in the same manner as with PET virgin resin.

2.5. Previous recycling efforts for CT/PET blends

Recovery of PET textile waste scrap has been studied extensively [18]. The polyester is either converted into starting polycondensation monomers, e.g., ethylene glycol and terephthalic acid or terephthalic acid esters, by alkaline hydrolysis [52] or by transesterification [7]; or it is recovered from mixed collections of common fibers using highly selective solvents for PET [56]. In the last case the common fibers are acetate, acrylic, CT, wool, nylons, polypropylene and rayon and the nature of solvents varies from chlorinated solvents/organic acids or phenol mixtures to aromatic polynuclear derivatives. Naphthalene has been found especially suitable for the practice of the separation process for dye stripping and as a primary dissolution solvent, because the solubility of PET in naphthalene is a strong function of temperature, ranging from zero solubility at 170°C to about 55% PET solubility

at 218°C (boiling point of naphthalene). In another method for PET recovery and reuse, textile waste formed of blended cellulose and PET fibers is treated with a mineral acid, such as H_2SO_4, under conditions which serve to hydrolyze the cellulose and convert it to a form which is readily removed from PET fibers while leaving the PET fiber substantially unaffected [27].

Some patented methods also consider the parallel recovery and recycling of cellulosic materials from different textile blends containing PET and cellulosic fibers. A recent process is described for recycling PET and CT blend fabrics by separating the PET from the CT fabric [55]. The process includes the following steps: charging the blend into a sulfone solvent, dissolving the PET at 190°C for a period of 35 minutes or less, separating the dissolved PET and sulfone solution from the CT fabric, filtering the CT to obtain isolated CT fabric, releasing the dissolved PET into a coagulation solution of a second sulfone solvent and water at which time the PET precipitates out of solution, and filtering the PET. A preliminary step is, however, necessary in which the fabric samples are dried at an appropriate temperature, pressure, and time interval to prevent degradation of the PET during the process.

Alternatively, textile waste composed of blended PET and cellulose fibers might be subjected to the action of glacial acetic acid and acetic anhydride in the presence of a catalyst under conditions that serve to convert the cellulose component of the waste to cellulose acetate. The acetate can be separated from the unreacted PET component in the form of a solution for a

manufacture of cellulose derivatives, whereas the PET is recovered in a form which may be garnetted to obtain a staple fiber for reuse [26].

2.6. Thermal analysis

Thermal analysis is a general term covering a group of related techniques whereby the dependence of the parameters of any physical property of a substance on temperature is measured. In differential thermal analysis (DTA) and differential scanning calorimetry (DSC) the occurrence of a temperature difference ΔT between the sample and the reference is the primary effect due to a thermal event within the sample.

In the case of DTA this ΔT is detected with a differential thermocouple and the resulting electrical signal is plotted as a function of time. The signal in a DSC experiment is related to the difference between the thermal response of the sample and the material in a reference cell as the two are heated or cooled at the same rate. In the scanning operation the sample and the reference show different temperature independent heat capacities [53]. A greater heat flow (dQ/dt) into the sample with the higher heat capacity is required to maintain a steady-state heating rate. The heat capacity at constant pressure (C_p) of a material is an extremely important thermal property. It provides direct experimental information on the possibilities of motion of the molecules and parts thereof. Quantities derived from it, entropy and enthalpy (H), can provide important information about the state of the material. From a technological point of view, quantitative heat capacity and enthalpy data are important. The usefulness of enthalpy can be shown by considering C_p. The definition of the

heat capacity at constant pressure can be written as the partial derivative of H with respect to temperature T, i.e., $C_p = (\partial H/\partial T)$. This property of a substance is defined as the temperature increase of a unit of substance as a result of the supply of a unit of heat. It is a very important property of a substance because it gives an indication of how energy is assimilated by the substance.

The temperature dependence of the quantities just mentioned is particularly important in crystallization and melting processes of semicrystalline polymers, as these often cover wide temperature ranges, and it is also a factor in the study of glass transition phenomena taking place in more or less amorphous polymers. The detailed information on glass transition, crystallization and melting, is critically important in formation, processing and utilization of fibers.

2.7. Rheology

In dynamic mechanical analysis, DMA, a sinusoidal strain or stress is applied to a sample and the response is monitored as a function of frequency [54]. The shear stress σ and the deformation (strain) γ are related via the shear modulus G as follows:

$$\sigma = G\gamma \qquad \text{(eq 1)}$$

The shear modulus is often known as the rigidity. Generally the measurements are represented as a complex modulus G^* to insure an accurate expression:

$$G^* = G' + iG'' \qquad \text{(eq. 2)}$$

where: $i^2 = -1$

If stress $\sigma(t)$ is applied, then altered with time t and angular frequency ω, expressed in radians per second, according to

$$\sigma(t) = \sigma_o \sin \omega t \qquad \text{(eq. 3)}$$

where σ_o is the amplitude. If the sample is an ideal energy elastic body, then deformation instantly follows the applied stress, and for this material,

$$\gamma(t) = \gamma_o \sin \omega t \qquad \text{(eq. 4)}$$

Polymers or their concentrated solutions, however, are viscoelastic materials, not ideal energy elastic bodies. In such cases of a controlled stress experiment the deformation lags behind the applied stress as shown in Figure 1. With ideal viscoelastic bodies, the resulting phase angle δ in the corresponding vector diagram can be assumed constant, such that the deformation or the strain is

$$\gamma(t) = \gamma_o \sin(\omega t - \delta) \qquad \text{(eq. 5)}$$

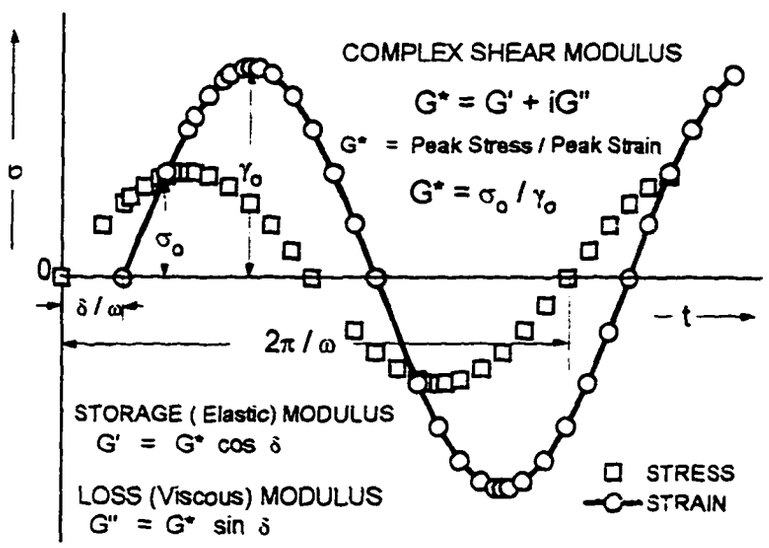


Figure 1. Schematic representation of the stress σ as a function of time t with the dynamic (sinusoidal) loading (strain) γ.

In general, the stress vector can be similarly considered to be the sum of two components. One component in a controlled strain experiment, that is $\sigma' = \sigma_o \cos \delta$, is in phase with the deformation, the other component, on the other hand, $\sigma'' = \sigma_o \sin \delta$, is not. A modulus can be assigned to each of these two components. The real modulus, or storage modulus, G', measures the rigidity and resistance to deformation of the sample. It is related to the complex modulus of rigidity G* by

$$G' = \sigma' / \gamma_o = (\sigma_o / \gamma_o) \cos \delta = G^* \cos \delta \qquad \text{(eq. 6)}$$

The imaginary, or loss modulus, G", on the other hand, reflects the loss of useful mechanical energy through dissipation as heat. Similarly, G" is given by

$$G'' = \sigma'' / \gamma_o = G^* \sin \delta \qquad \text{(eq. 7)}$$

Introducing the complex variables, one may rewrite eqs. 3 and 4 as follows:

$$\sigma^* = \sigma_o e(i\omega t) \qquad \text{(eq. 8)}$$

and

$$\gamma^* = \gamma_o ei(\omega t - \delta) \qquad \text{(eq. 9)}$$

The complex modulus G* may be then expressed as shown in eq. 2.

In a stress controlled instrument (such as the Seiko DMS110 module) the force is transmitted in accordance with the generated sine wave to the specimen, through the probe, by means of the function generator. The strain produced in the sample is detected by a differential transformer and produces a signal that expresses the dynamic viscoelasticity of the sample from the force and the strain. This is termed stress controlled testing, in contrast to the strain controlled DMA, in which a sinusoidal varying stress is applied and the strain response is measured.

In reporting the results of DMA testing, use is often made of the two other parameters derived above, i. e. the storage and the loss moduli, G'(w) and G"(w), respectively, and the loss tangent, or tangent of the phase angle, δ, which indicates the lag in the response compared to the applied strain or stress. For purely elastic materials, the phase angle will be zero, whereas for purely viscous materials, the phase angle will be 90°. Thus, the phase angle, expressed as its sine or tangent, is an important parameter for describing the viscoelastic properties of a polymeric material. The loss tangent is calculated simply as the tangent of the phase angle, or alternatively, as the ratio of the loss to storage moduli (Figure 2):

$$\tan \delta = G'' / G' \qquad \text{(eq. 10)}$$

and

$$G^* = [(G')^2 + (G'')^2]^{1/2} \qquad \text{(eq. 11)}$$

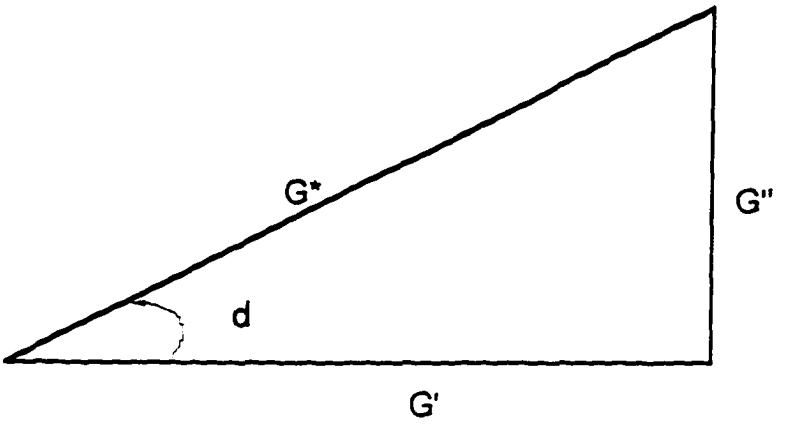


Figure 2. Relationships among dynamic moduli and the phase angle shown schematically through the trigonometry of a right triangle.

In analyzing polymeric materials (or their concentrated solutions), G*, the ratio of the peak stress to the peak strain, reflects the total stiffness. The in-phase component of [G*], i. e. the shear storage modulus, [G'], represents the part of the input energy which is not lost to heat (the elastic portion). The out-of-phase component of [G*], i. e. the shear loss modulus, [G''], represents the viscous component. The complex dynamic shear viscosity η can be obtained from G* divided by the frequency, while the dynamic viscosity is $\eta = G''/\omega$. Therefore, at temperatures at least 20°C above the Tg, any of these three parameters should relate to flow under load. In the present study η and tangent δ were selected to evaluate the viscous flow of cellulosic solutions under investigation.

3. RESEARCH METHODOLOGY

3.1. Materials

Plain weave 100% CT and 100% PET fabrics with no finishing treatment were used for the hydrolysis of PET. A variety of colored fabrics at different blend ratios of CT and PET were purchased in a fabric store and used as actual samples for the recycling processes. A discarded CT/PET shirt was also included as an example in this study. Only PET fabrics, such as Dacron, commonly manufactured by reacting terephthalic acid with ethylene glycol, were used throughout the study.

NMMO was purchased from Aldrich as a monohydrate or as a 50% solution in water. Solutions of NaOH of various concentrations were prepared before use from pellets. Anti-oxidant, n-propyl gallate, was used to reduce the degradation of cellulosic molecular chains in the process of making cellulosic solutions from the recovered CT. A commercial bleaching agent, sodium hypochlorite (NaOCl), was purchased and diluted to specific concentrations for bleaching process. A direct dye, C.I. Red 81, and a reactive dye, C.I. Blue 163, were used for dyeing lyocell fibers.

3.2. Methods for determining fiber composition of CT/PET blends

Thermal methods were used for determining blend ratios. All data were collected in a nitrogen atmosphere using SEIKO DSC 220C and SEIKO

TG/DTA 220 instruments. Heating rates, of 5°C/min and 2°C/min were used for DSC and TG/DTA determinations, respectively. Indium and tin of 99.9+% purity were used for calibration of both instruments at the respective heating rate. The melting temperature (T_m) was identified as the first endothermic deflection of the base line, i.e., as the intersection of the base line with the extrapolated tangent to the left arm of the melting peak, while the temperature at the peak was identified as T_{max} . Accepted literature values for T_m (T_m^{In}=156.6°C and T_m^{Sn}=232.0°C) and ΔH_m (ΔH_m^{In}=28.6 mJ/mg and ΔH_m^{Sn}=60.6 mJ/mg) were used for computations [52]. Samples of known blend ratios were prepared either by finely cutting PET and CT swatches (with a scissors), and mixing them in the desired ratio or by finely grinding selected scissored blends (in a coffee grinder) to accomplish blending at the fiber level. Commercial plain CT, PET and CT/PET or rayon(R)/PET bicomponent fabrics were also analyzed as scissored or ground samples. R/nylon 6,6 (N) composite fibers were analyzed as cut samples. All samples were conditioned at 70°F (21°C) and 60% relative humidity before use.

3.3. Separation of textile components from CT/PET blends

3.3.1. Separation method 1: hydrolysis of PET in alkaline solution

CT/PET blended fabrics of different blend ratios were used for separation of CT and PET. The PET component was subjected to alkaline hydrolysis in different concentration NaOH solutions at temperatures between 95-108°C. Ethylene glycol was added as a wetting agent for PET. Terephthalic acid (m.p. 321.8°C as determined with DSC, ΔH=750mJ/mg) was subsequently

precipitated quantitatively by acidifying the solution with H_2SO_4 after the removal of the CT residues by filtration. The liquid part obtained after the filtration of terephthalic acid was further concentrated in a Buchi rotavapor at 100°C as an ethylene glycol aqueous solution.

The CT fabric pieces were washed with distilled water and line dried. Subsequent bleaching was carried out in colored fabrics to improve the whiteness of the CT residues obtained from the alkaline treatment. NaOCl was used as a bleaching agent at different concentrations at room temperature. The bleaching of CT/PET blends and 100% CT fabrics without alkaline treatment was also carried out for comparison purposes. The lightness (L*) of various bleached fabrics with or without alkaline treatment was measured by using a Pacific Scientific colorimeter according to AATCC test method 153-1985 [4]. The breaking strength of CT was determined using an Instron 4301 tester according to the ASTM D-1682-64 cut strip method [5].

3.3.2. Separation method 2: dissolution of CT in NMMO·H_2O

CT/PET blended fabric swatches were placed in a flask with NMMO·H_2O at 85-90°C at a ratio of 1-2% on the weight of bath. The PET component was removed by filtration at 85°C, washed with hot NMMO.H_2O, then washed with distilled water, and dried. Subsequently, it was submitted to the first hydrolysis step together with a new fabric sample. The cellulose containing filtrate was concentrated to a spinnable 10-17% cellulose solution. High grade wood pulp was also tested as an additional cellulose source.

3.4. Preparation of cellulose solutions

A typical example for the preparation of 15% cellulose solutions for fiber extrusion is as follows. To a 500cc spherical flask containing 148g of 50% aqueous NMMO were added 148g water. The flask was immersed in a water bath and the bath temperature was increased to 75°C. The separated cellulose materials from blended fabrics were cut and ground finely in a coffee grinder. A slurry was then obtained by adding 15g of the finely ground cellulose (The amounts of NMMO, water and cellulose were modified to the concentrations). The flask was connected to a Buchi rotavapor operated at 70°C and atmospheric pressure. The temperature of the bath was increased to 100°C and the rotavapor was then connected to a water pump giving a vacuum. Within 1 to 2 hrs of rapid rotation, a brownish film of transparent cellulose solution was observed on the inner wall of the flask as water was gradually removed by evaporation. The final solution is composed of cellulose, NMMO and water, the last two components being in a 1:1 molar ratio. The film was bubble free and had a thickness of between 2.5 and 5mm. The absence of bubbles and insoluble particles was checked by sampling a drop of solution and positioning it on a glass slide for observation with a polarizing optical microscope. The flask was then brought to atmospheric pressure, cooled down to room temperature, capped, and refrigerated until further use.

3.5. Rheological characterization of cellulose solutions

Rheological characteristics of 10-15% cellulose solutions in NMMO monohydrate (NMMO·H_2O, m.p.. 72°C by DSC at 5°C/min) were determined

using a Seiko dynamic mechanical spectrometer DMS-110 and an advanced capillary extrusion rheometer (ACER) made by Polymer Laboratories (now Rheometrics).

3.6. Spinning of lyocell fibers

The ACER (Figure 3) was used as an extruder to spin lyocell fibers from the cellulosic solution in NMMO·H_2O. The ACER is equipped with a pressure transducer. A specially designed die with a 500μm one hole spinneret was used for spinning fibers into a hot water bath.

3.7. Dyeing of lyocell fibers

3.7.1. Dyeing lyocell fibers with a direct dye

Dyeing with a direct dye, C.I. red 81, was carried out according to the standard procedure for direct dyes [10]. Two percent dye on the weight of fibers (owf) and 20% salt owf were included in the dyeing solution. The liquor ratio was 40 to 1. The dye bath with wet fibers was heated to boiling, then the temperature of the dyebath was reduced, and maintained at simmer for 15 mins. Dyed fibers were rinsed with cold and hot water to remove unreacted dye, and they were dried in the air. The cross section and the longitudinal appearance of the dyed fibers were observed with a polarizing optical microscope.

3.7.2. Dyeing lyocell fibers with a reactive dye

Dyeing with a reactive dye, C.I. blue 163 was carried out according to the standard dyeing procedure [9]. Two percent dye owf in a liquor ratio of 20 to 1 was used in the dyebath. Thel steps of the standard dyeing procedure are

shown in Figure 4. The cross section and the longitudinal appearance of the dyed fibers were observed with a polarizing optical microscope.

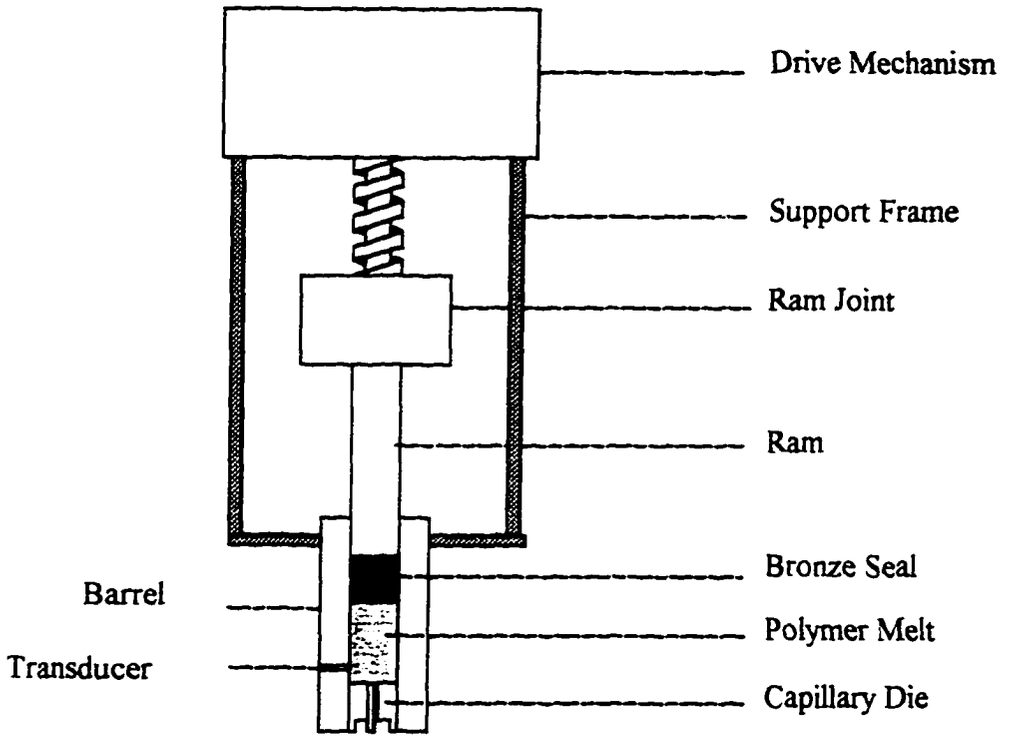
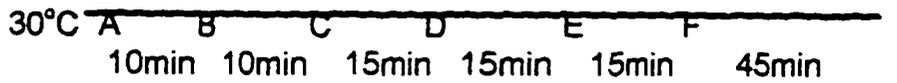


Figure 3. Diagram of an ACER instrument.



A: Dye 2% owf
B: NaCl 2.5g/L
C: NaCl 7.5g/L
D: NaCl 25g/L
E: dissolved Na_2CO_3 0.2 g/L
F: dissolved Na_2CO_3 3.8 g/L

Figure 4. Standard dyeing procedure with a reactive dye.

4. RESULTS AND DISCUSSION

4.1. Determination of fiber content of CT/PET blends with thermal analytical methods

The temperature of melting of the crystalline fraction (T_m) and the associated enthalpy change (ΔH) are the two parameters on which the identification and quantification of semicrystalline polymers are based when analyzed by thermal methods. The ΔH during PET melting, ΔH_{PET}, was determined by DSC using ten samples of 100% PET fabrics. T_m, T_{max}, and the corresponding ΔH are listed in Table 1. Both T_m and T_{max} are listed since the latter is more readily distinguishable than T_m. For the sake of clarity, the DSC thermograms of the first five samples only are presented in Figure 5.

Table 1. Thermal characteristics of 100% PET fabric samples: T_m, T_{max}, and the corresponding ΔH_{PET}, as determined by DSC. Heating rate, 5°C/min.

Sample No.	Weight, mg	T_m, °C	T_{max}, °C	ΔH_{PET}, mJ/mg
1	5.6	252.5	256.0	54.0
2	4.8	251.2	255.2	53.3
3	5.1	252.5	255.7	53.4
4	5.2	252.1	256.4	54.6
5	5.8	252.3	256.4	54.1
6	4.5	252.6	256.2	54.8
7	5.9	252.1	256.3	54.9
8	4.8	251.9	256.0	55.2
9	5.2	252.3	256.2	53.1
10	5.0	252.1	255.9	54.0

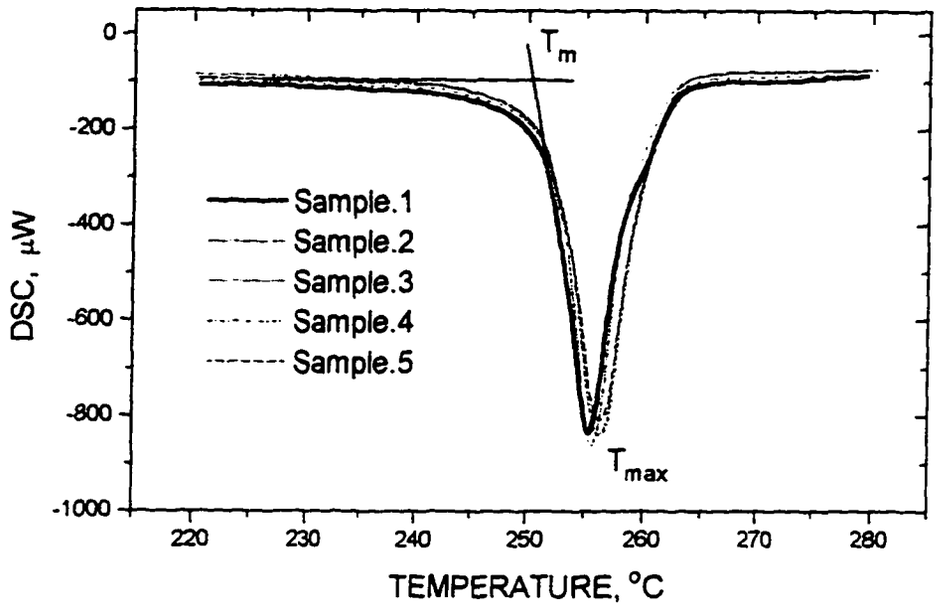


Figure 5: DSC thermograms of five different 100% PET fabric samples.

The mean value and standard deviation of ΔH's in 100% PET samples were 54.1mJ/mg and 0.7 mJ/mg respectively. Similar data were obtained by DTA, with a mean value for ΔH_{PET} of 54 μV.s/mg. However, the T_m =249.3°C registered in this case was lower than that determined by DSC due to the lower thermal rate used for heating, i.e., 2°C/min instead of 5°C/min.

Ten samples of CT and PET blends were prepared by weighing finely cut pieces of CT and PET fabrics and were analyzed by DSC (Figure 6) and DTA (Figure 7). The percent content of PET in blended fabrics, %PET, was confirmed using the following equation:

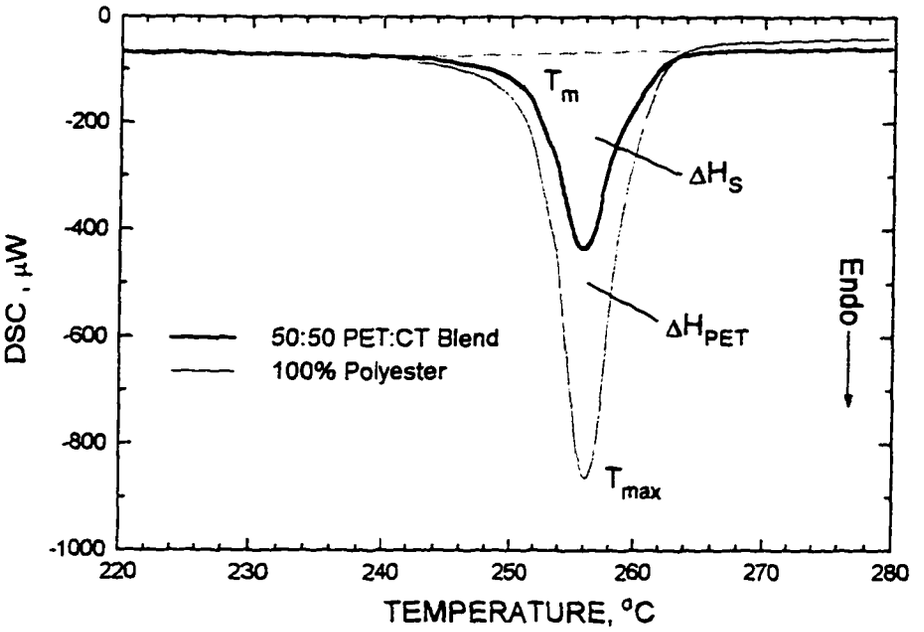


Figure 6. DSC thermograms of 100% PET fabric and of a blend of CT/PET scissored fabrics in a 50:50 weight ratio.

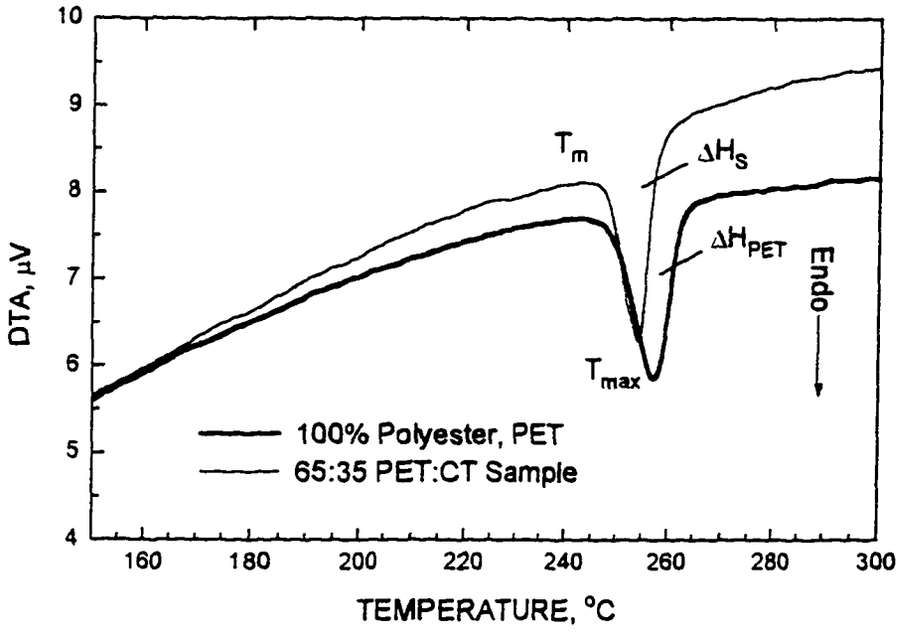


Figure 7. DTA thermograms of 100% PET fabric and of a blend of CT/PET scissored fabrics in a 35:65 weight ratio.

$$\%PET = (\Delta H_S \times 100)/\Delta H_{PET} \qquad \text{Eq. 1}$$

where:

ΔH_S is the enthalpic change during the melting of PET from blended samples determined by DSC (and expressed in mJ/mg) or by DTA (and expressed in μV.s/mg), and ΔH_{PET} is the enthalpic change during the melting of a 100% PET sample determined by DSC (in mJ/mg) or by DTA (in μV.s/mg).

The percent content of CT, %CT, was calculated taking into account the amount of water absorbed by CT as determined by thermogravimetry, i.e., the weight loss read on the corresponding TG curve at 150°C in Figure 8:

$$\%CT = 100 - (\%PET + \%W_{H2O}) \qquad \text{Eq. 2}$$

where $\%W_{H2O}$ is the weight loss at 150°C. The results are listed in Table 2. For comparison purposes, this table contains the fabric contents of CT without considering the absorbed water, $\%CT_{H2O}$, calculated as the difference from one hundred percent:

$$\%CT_{H2O} = 100 - \%PET \qquad \text{Eq. 3}$$

The "dry" CT is the important content of CT/PET blends in recycling the cellulose as a new fiber by solubilization in NMMO·H_2O because the concentration of water is critical for cellulose solubilization.

The good agreement between the actual fiber content in mixed samples and the fiber content determined by thermal analysis was confirmed by parameter estimation in a simple regression model. Using the data listed in Table 2, a significant linear relationship between DSC readings and the PET content in mixed samples was found with a probability p-value of 0.0001 and R-.

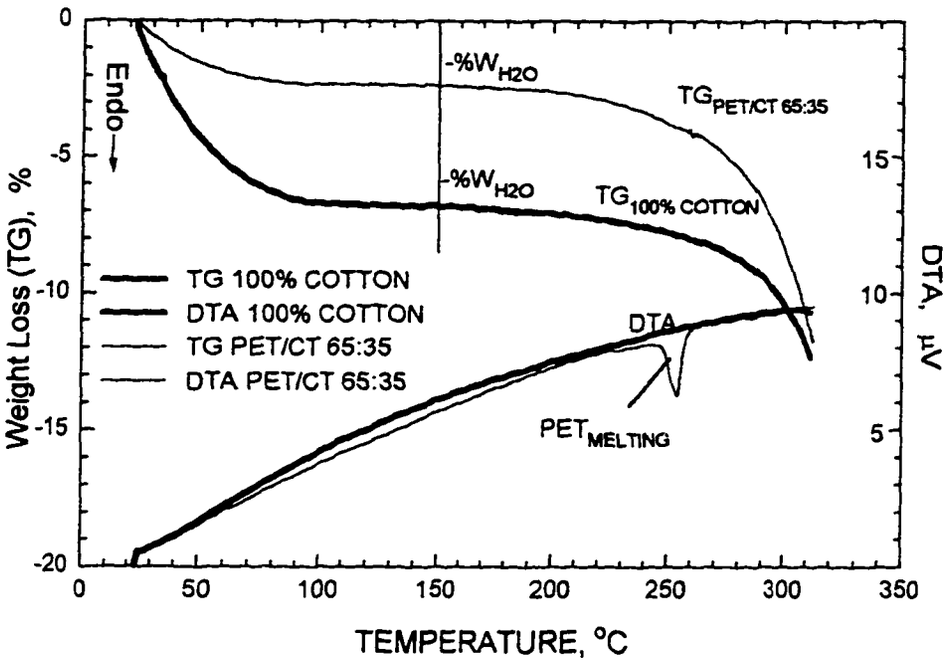


Figure 8. TG and DTA thermograms of 100% CT fabric and of a blend of CT/PET scissored fabrics in a 35:65 weight ratio.

Table 2. Comparison between the actual content of CT and PET of blends prepared from scissored plain CT and PET fabrics and the content of blends determined by DSC and TG/DTA using eqs. 1-3.

Sample No.	Actual PET:CT	ΔH_{PET} mJ/mg	%PET from eq.1	%CT from eq.2	$\%CT_{H2O}$ from eq.3
11	85.2 : 14.7	47.5	87.8	11.5	12.2
12	67.7 : 33.3	37.4	69.1	29.3	30.9
13	65.6 : 34.4	35.3	65.2	33.1	34.8
14	58.6 : 41.4	29.4	54.3	43.6	45.7
15	58.1 : 41.9	30.8	56.9	41.0	43.1
16	51.8 : 48.2	28.0	53.2	44.4	46.8
17	41.7 : 58.3	23.2	42.9	54.2	57.1
18	28.8 : 71.2	15.0	27.7	68.8	72.3
19	17.7 : 82.3	8.6	15.9	80.1	84.1
20	10.0 : 90.0	6.5	12.0	83.4	88.0

square of 0.99. Therefore, an accurate blend ratio of CT and PET blends can be predicted directly from the ΔH during melting of PET in DSC or DTA thermograms.

Table 3. Comparison between the actual content of CT and PET of blends prepared from plain CT and PET fabrics ground together at the fiber level and the content of blends determined by DSC using eq.3.

Sample No.	Actual PET:CT Ratio	ΔH_{PET}, mJ/mg	DSC PET:CT Ratio
21	73 : 27	38.2	70.8 : 29.2
22	73 : 27	38.5	71.2 : 28.8
23	73 : 27	39.7	73.4 : 26.6
24	73 : 27	40.4	74.7 : 25.3
25	73 : 27	38.9	71.9 : 29.1
26	73 : 27	39.9	73.7 : 26.3
27	73 : 27	40.2	74.3 : 25.7
28	73 : 27	40.4	74.7 : 25.3
29	73 : 27	39.5	73.0 : 27.0
30	73 : 27	39.8	73.6 : 26.4

Additional mixtures were prepared by grinding known amounts of CT and PET fabrics in order to achieve blending at the fiber level. The blend ratios of the samples were then checked by DSC. The results for ten samples containing 73% PET are presented in Table 3. The mean, standard deviation and the coefficient of variation of PET percent in these fabric blends were 73.1%, 1.4% and 1.9%, respectively. For another series of ten samples containing 50% PET (No. 31-40), the mean, standard deviation and the coefficient of variation in the experimentally determined PET content in the fiber form were 48.8%, 1.5% and 2.9% respectively. The slight differences in the PET content as determined by DSC vs. the actual content were attributed to experimental errors, such as weighing small amount of short fiber specimens, forming homogeneously blended samples, and absorption of water while handling the samples.

The thermal analysis procedure was applied to samples of purchased fabrics of PET and CT or R blends and results compared to labeled fiber contents (Table 4). Not only were some compositions significantly different from

Table 4. Comparison between the blending ratio of CT and PET labeled on different commercial products and the ratio determined by DSC.

Sample No.	Labeled %PET : %CT	ΔH_{PET} mJ/mg	%PET from eq.1	$\%CT_{H2O}$ from eq.3
X1	0 : 100	0.0	0.0	100.0
X2	0 : 100.	28.9	53.4	46.6
X3*	30 : 70	25.6	47.3	52.7*
X4*	50 : 50	27.7	51.2	48.8*
X5	50 : 50	29.3	54.2	45.8
X6	50 : 50	33.2	61.4	38.6
X7	50 : 50	33.3	61.6	38.4
X8	65 : 35	34.7	64.1	35.9
X9	65 : 35	38.9	71.9	28.1
X10	100 : 0	54.3	100.4	0.0

*) % Rayon instead of %CT

the labeled ratio, but some CT/PET blends which were advertised as 100%CT fabrics, such as X2, were found to be blends with PET. Solubility and microscopic tests confirmed the presence of PET in the warp and CT in the filling [3]. Examples of thermal analysis for other commercial fabrics are included in Appendix A. The students who bought fabric samples are identified in their thermograms.

This method was also applied for determination of the R content in generic bicomponent R/N sheath/core fibers produced experimentally in the laboratory. From ΔH during melting of 100% N fibers, ΔH_N, and that of R/N fibers, $\Delta H_{R/N}$, (Figure 9), the content of N core in the bicomponent fibers was

calculated as 78.4% using Eq.1, where ΔH_N was substituted for ΔH_{PET} and $\Delta H_{R/N}$ was substituted for ΔH_S. Table 5 gives the thermal values obtained. The water content determined from TG data at 150°C was 4.6% and represented the water absorbed both by R and N. The water content determined in the same way for a 100% N sample was 2.3%. Therefore, the water absorbed by the R sheath was 2.8%, or, 16% of the dry cellulose content. This water absorbency was high for

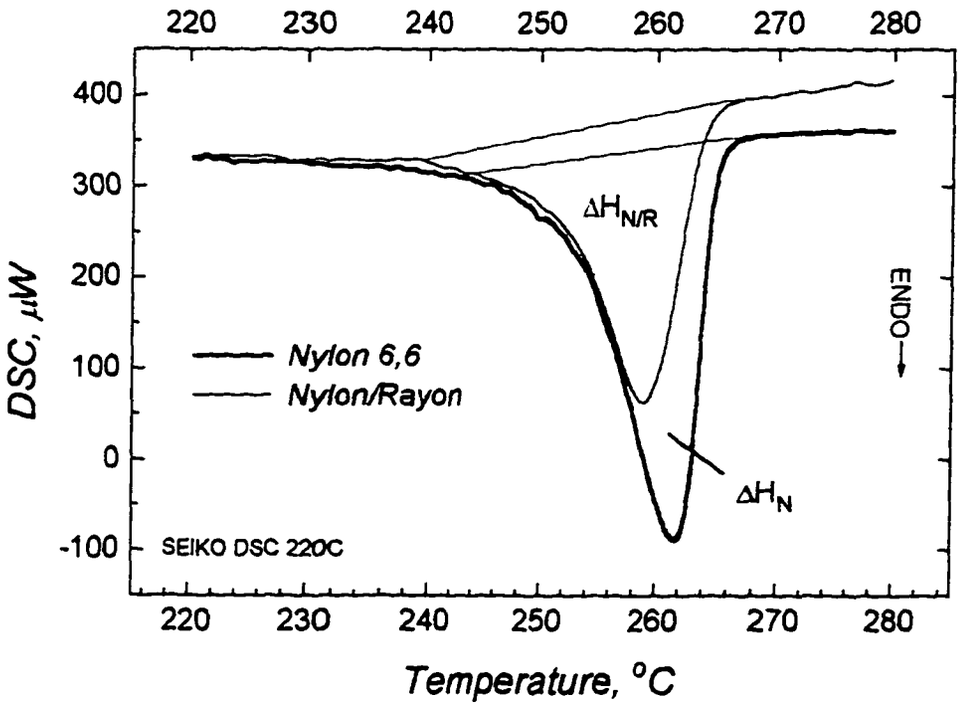


Figure 9. DSC thermograms of 100% N fibers and of generic bicomponent R/N 6,6 sheath/core fibers.

R, but it should be taken into consideration that the cellulose sheath covering the N core might be highly amorphous because no orientation of chains was imposed during cellulose regeneration [16,59].

Table 5. Determination of R and N contents of bicomponent R/N sheath/core fibers from DSC and TG data.

Fiber	ΔH, mJ/mg (DSC)	-%W_{H2O} at 150°C	% Nylon 6,6 (dry)	% Rayon (dry)
Nylon 6,6	45.4	2.3	100	0
Rayon/Nylon	35.6	4.6	78.4	17.0

4.2. Hydrolysis of PET component in CT/PET blends

A mixture of 100% CT and 100% PET fabric swatches was used for complete hydrolysis of PET in the initial experiment. In order to determine the time required for complete hydrolysis of PET fabrics, the mixture of fabric swatches was placed in a beaker with a glass cover. The glass cover was used to minimize the evaporation which was generated at the temperature of over 103°C. Thinning and breaking up of PET fabric pieces was observed in the hydrolysis of PET. The complete dissolution of PET fabric swatches was visible evidence for the complete hydrolysis of PET in this initial experiment. The molarity of NaOH versus the time for complete hydrolysis of PET fabrics is plotted in Figure 10.

For the lower temperature range of treatment (at 95-100°C), the hydrolysis time for PET was longer. At NaOH concentrations over 4.0M, however, there is little difference in the time for complete hydrolysis of PET fabrics. Vigorous boiling of the solution was observed in the temperature range of 103-108°C.

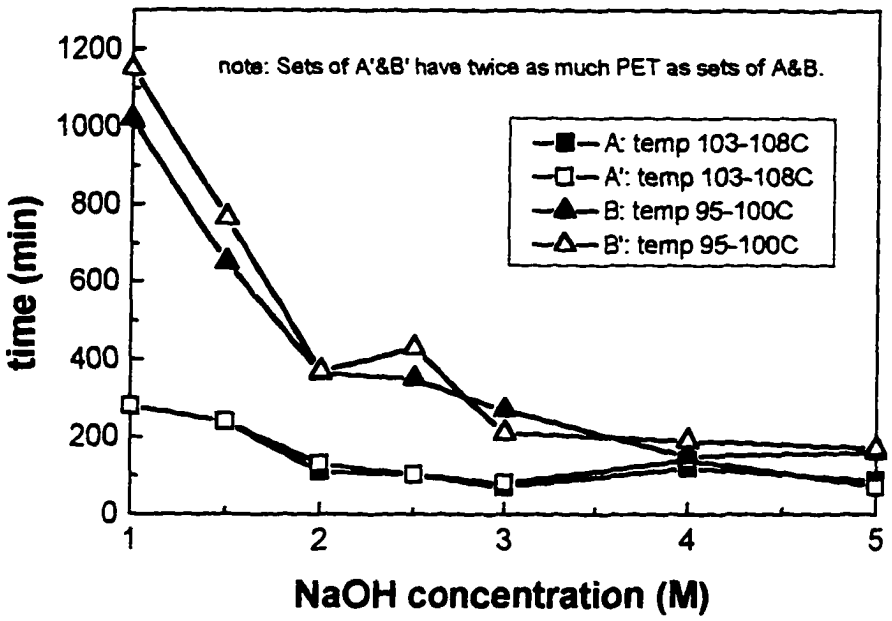


Figure 10. Time for complete hydrolysis of PET at different NaOH concentrations.

As a concern in the study was maintenance of the high DP in the cellulosic chains, the breaking strength of CT was examined as an indirect indication for any change in DP. The lower concentrations of NaOH and a lower temperature range tended to result in longer periods of time for complete hydrolysis, which explained the reduction in the breaking strength of the relating CT. An inspection of Figure 11 shows, however, that no significant loss of breaking strength of CT fabrics occurred when the hydrolysis was carried out, even for 280 min (Figure 10) at 103°C and 1M NaOH concentration, or 70 min (Figure 10) at 108°C and 5M NaOH concentration. This suggested that a wide range of hydrolysis parameters (alkaline solution concentration and

temperature) could be considered for economic optimization of reaction parameters.

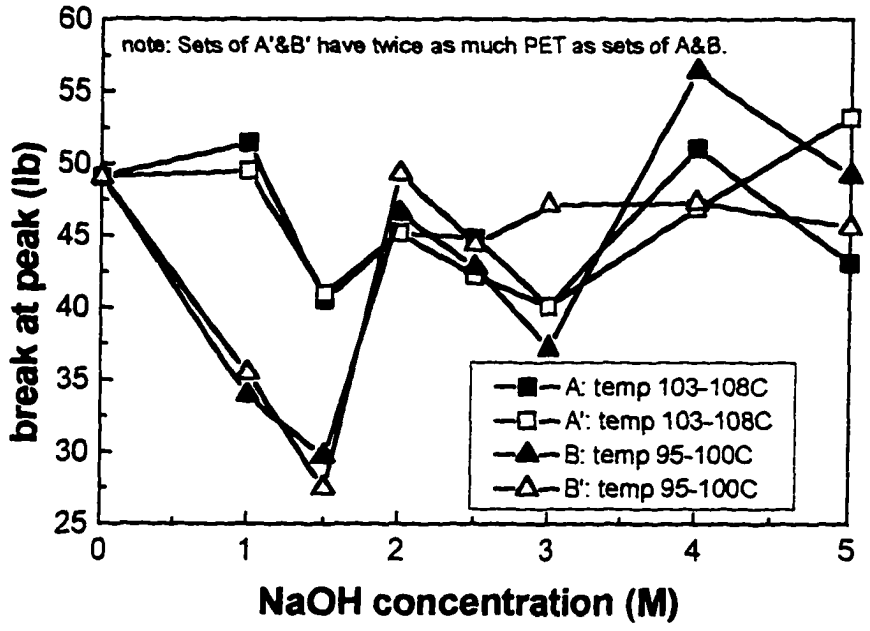


Figure 11. Breaking strength of CT residues in the different concentrations of NaOH solutions for hydrolysis of PET.

However, significant loss of breaking strength was shown at the lower NaOH concentrations between 1M-3M at the lower temperature range. This implies that alkaline treatment for a long period of time negatively affected the cellulosic molecular chains in CT. On the other hand, the breaking strength of CT was not changed at the higher NaOH concentrations which led to the shorter time for the complete hydrolysis of PET. As the longer hydrolysis time of PET might result in the higher consumption of energy, the concentration of

4M NaOH and the temperature of 103°C were selected for the condition parameters for complete hydrolysis of PET fabrics in CT/PET blends.

Slightly higher breaking strength of some CT samples was noticed with treatments of 4M and 5M NaOH solutions at the higher temperature range of 103-108°C, probably due to an effect similar to slack mercerization. In general, mercerization is carried out with 14-32% NaOH solution at certain temperature and for a short period of time, which results in the swelling of the crystalline regions as well as the amorphous regions of cellulose and reduced crystalline size. The mercerization includes the transformation of the fiber pattern, especially the crystalline region, from cellulose I to cellulose II. In addition to mercerization effect, the hydrolysis of PET in the alkaline treatment seemed to be beneficial for the following bleaching step in the recycling of CT/PET blends. The alkaline treatment lead to the mercerization effect in CT causing increased accessibility to the fine structure of the CT component.

The thermal analytical methods of DSC and TG/DTA were used for confirming the complete hydrolysis of the PET component in the case of actual recycling process for the CT/PET blends. As mentioned in section 4.1, the DSC thermogram of the fabric samples was checked before filtering the CT residue out of the hydrolyzed PET filtrate. The exemplary DSC thermograms are included in Appendix B.

4.3. Bleaching

4.3.1. Bleaching of CT/PET blends without alkaline treatment

Bleaching (or dye-stripping) for recycling is an optional step as the color of the blended fabrics may be used as it is for the specific end-uses of the recycled products. This work, however, attempted to determine whether the optional bleaching step would affect the DP of the CT component which would be subsequently used for making cellulosic solutions for spinning fibers. The aim of bleaching in the process of textile recycling is to improve the dyeability of a recycled CT fiber by ensuring a high and uniform degree of whiteness which would result in better quality recycled fibers. NaOCl was selected as a bleaching agent because it required lower concentrations, no heating, and is readily available at low cost.

Colored fabric samples, 100% CT or CT/PET blends, showed no significant change in color when bleaching was carried out at room temperature without any pretreatment. Figure 12 shows the lightness of the CT/PET blends after bleaching with 2% NaOCl solution at different time intervals. All 100% CT fabrics of two different colors were used for comparison purposes. A navy blue 100% CT did not show any visible change in bleaching either because of a finishing treatment for color resistance to bleaching or dyeing with a chlorine resistant dyestuff. A significant change in color was, however, observed in another pink 100% CT fabric after 30 minutes bleaching, which seemed to show the evidence of poor washfastness of a direct dye.

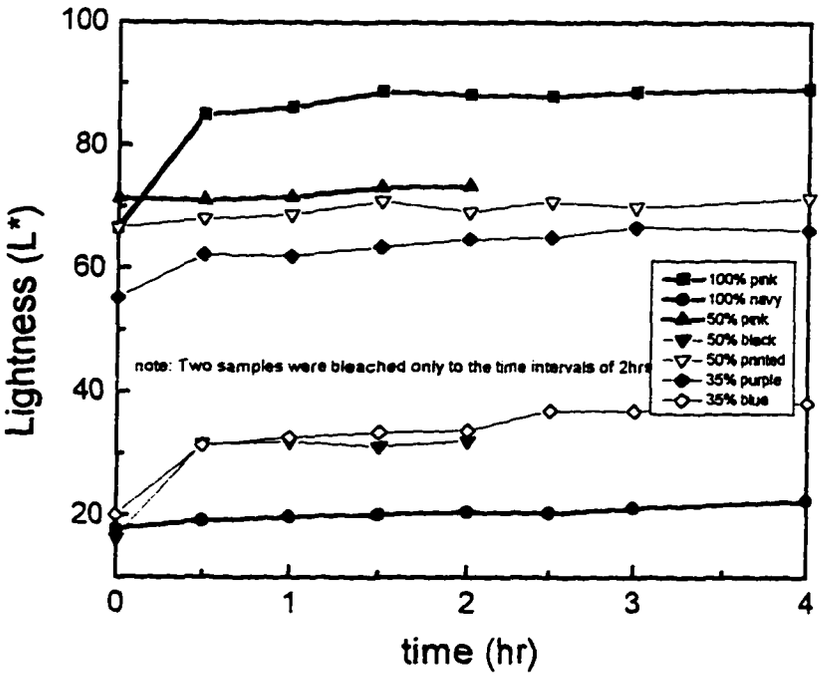


Figure 12. Lightness of bleached fabrics without alkaline treatment.

A color change was not visually observed in 65%/35% PET/CT blends. The CT content might have been too small to show noticeable change in color and the PET component dyed with disperse dye might not be affected by bleaching. However, a linear relationship between the lightness of CT/PET samples and bleaching time was found at the 0.05 level (Appendix C). Bleaching with 2% NaOCl solution for an hour was selected as an optimum condition for minimal change in the mechanical property of CT and maximum bleaching effect. As the recycling of the CT component was the main focus in the study, the bleaching conditions were determined by considering the resultant color change of 100% CT fabrics.

The breaking strength of bleached 100% CT and CT/PET blends was determined (Figure 13). The higher breaking strength of blends resulted from the higher composition of PET component in blends. The 100% CT fabrics lost most of their breaking strength in the first two hour of bleaching, with the strength loss decreasing after that. A significant effect of bleaching time on breaking strength was found for all fabrics (Appendix C).

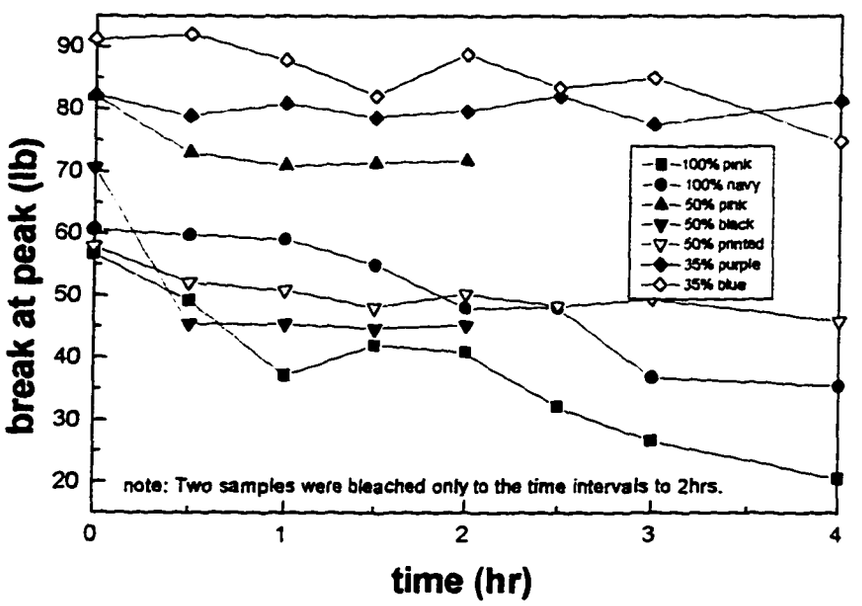


Figure 13. Breaking strength of bleached fabrics without alkaline treatment (warp).

Breaking strength of the CT component in blends decreased with bleaching, predicting the degradation in DP of CT because PET was generally known not to be affected by bleaching with NaOCl.

Bleaching was attempted before and after hydrolysis of PET for the CT/PET blends. The bleaching of CT/PET blends followed by alkaline

treatment caused severe destruction to fabrics, which resulted in great difficulty in the following separation stage. Therefore, the alkaline treatment for separation of PET from CT/PET blends was selected as the step prior to the optional bleaching step.

4.3.2. Bleaching CT residues of the CT/PET blends after alkaline treatment

Alkaline treatment at 4M NaOH solution was carried out for complete hydrolysis of the PET component in CT/PET blends of various colors. The CT components were filtered out, rinsed with distilled water thoroughly, and then line dried. There was no observable destruction of the fabrics during the alkaline treatment.

Alkaline treatment prior to bleaching with NaOCl provided the advantage of minimizing the loss of breaking strength of the CT component along with increasing absorptivity due to the slack mercerization effect mentioned above. It might also be beneficial to maintain a relatively high pH for the bleaching solution, preventing accidental lowering of the pH. The bleaching process with NaOCl sometimes causes severe damage to the DP of CT when the pH of the bleaching solution is low.

The effects of bleaching on fabric whiteness are shown in Figure 14. The -1 and 0 in the x-axis in Figures 14-16 represent the washed control of CT/PET blends and alkaline treated CT components after filtration of PET respectively. The remainder of the x-axis represents time intervals for bleaching. A significant increase in lightness of the separated CT residues and 100% CT fabric was obtained after 30 minutes bleaching with 2% NaOCl

solution. The high R-square in the statistical analysis also confirmed the significant differences in lightness of colored fabrics at different bleaching time (Appendix D). There was slight and gradual increase in lightness of CT residues at the increase of bleaching time after 30 minutes even though it was not visibly apparent. The visible increase in whiteness of the alkaline treated and bleached fabrics can be seen in the samples (Appendix E).

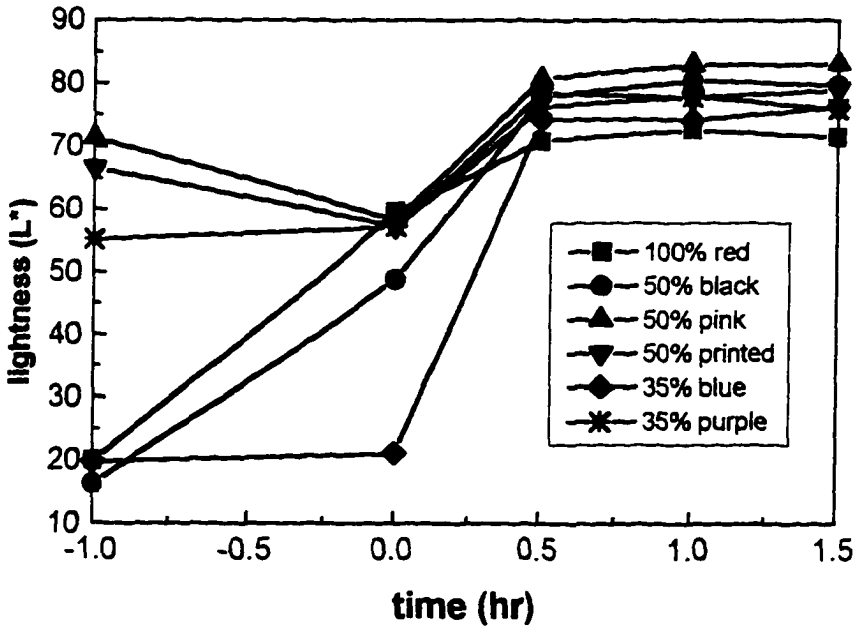


Figure 14. Lightness of alkaline treated and bleached fabrics.

The breaking strength of CT residues from the differently colored CT/PET blends was examined again as an indirect indication for any change in DP of cellulosic chains which might have been caused during the alkaline treatment and subsequent bleaching (Figures 15-16). The higher initial breaking strength of CT/PET blended, washed, control fabrics was attributed

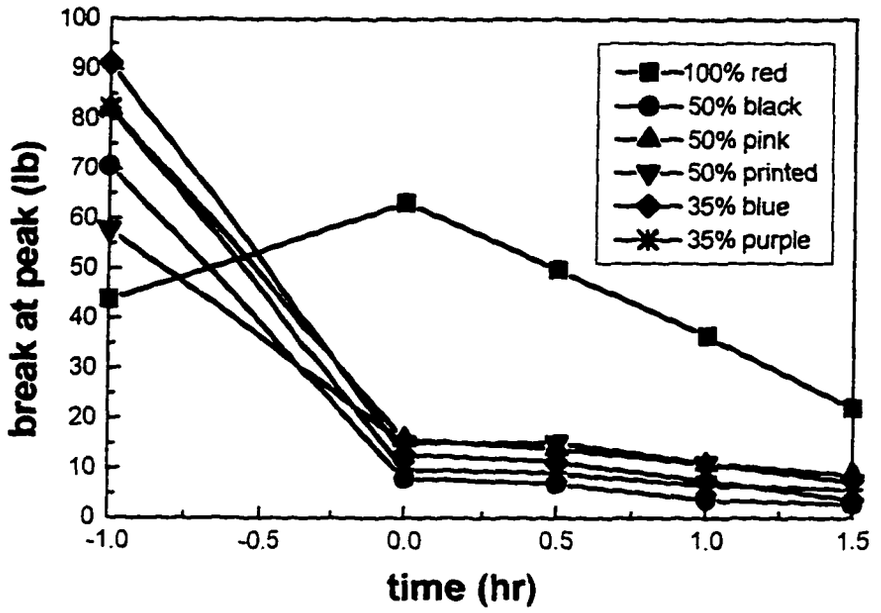


Figure 15. Breaking strength of alkaline treated and bleached fabrics (warp).

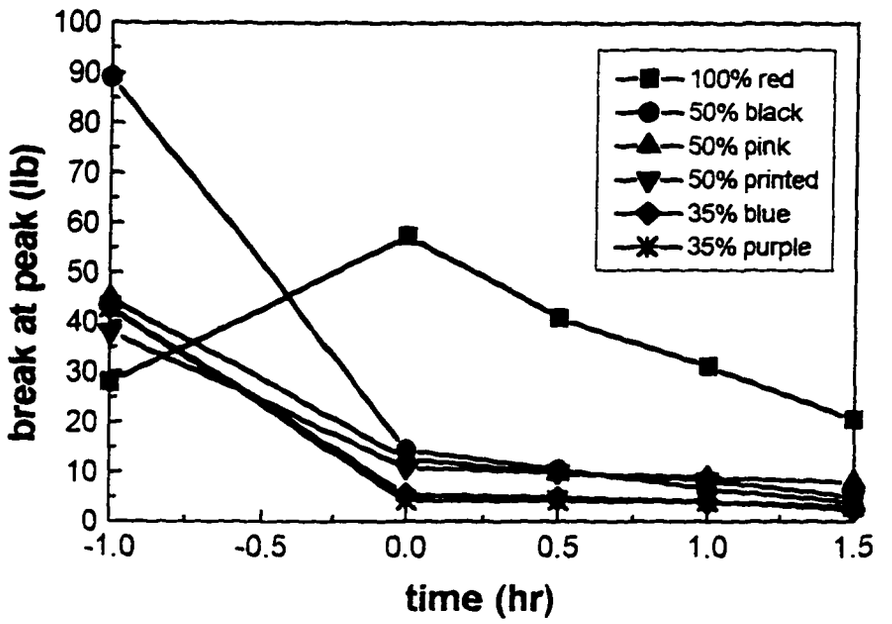


Figure 16. Breaking strength of alkaline treated and bleached fabrics (filling).

mainly to the percentage of PET composition in the blends. The significant loss of breaking strength in the fabrics was due to bleaching because it was apparent in the 100% CT samples. It is possible that the alkaline treatment prevented significant loss of strength in CT residues, and promoted bleaching of the CT.

As mentioned in the previous section, alkaline treatment at 4M NaOH solution resulted in an increase in breaking strength of the 100% CT fabric. Breaking strength of the blends decreased significantly due to separation of the PET component after hydrolysis. The separated CT components from the blends showed a gradual decrease in breaking strength over the course of the bleaching process, which was also confirmed in statistical analysis (Appendix D).

4.4. Rheological characterization of cellulose solutions and spinning of lyocell fibers

Five parameters are necessary in order to characterize rheologically the cellulose-NMMO solutions: the nature and the DP of cellulose, the temperature, the amount of water and the concentration of cellulose. The concentration of NMMO-cellulose solutions is an important factor for determining the experimental conditions (i.e., the temperature and the time) for achieving a complete dissolution of cellulose. When the dissolution occurs in the presence of water, there is competition between water and cellulose for complexing with NMMO molecules which seem to prefer water to cellulose. That is why cellulose solutions were prepared with a minimum water content, corresponding

to a monohydrate composition of the NMMO solvent . For this purpose, a dissolution temperature over 75°C was maintained.

The viscosity, η, of dilute cellulose solutions in many nonaqueous solvent systems is rather high due to the chain-stiffening effect of complex formation between cellulose and solvent. As shown in Figure 17, a steep viscosity increase was observed during solution formation due to the increase of cellulose concentration assisted by raising the temperature. An even steeper increase occurred after reaching 105°C caused by the isotropization of the cellulose-solvent system. Formation of a liquid crystalline phase by cooling a concentrated cellulose solution in NMMO·H_2O from high temperatures (e.g., 115°C) below 75°C was observed by polarized light microscopy (Figure 18). A nematic pattern was seen over a large temperature domain below 75°C before

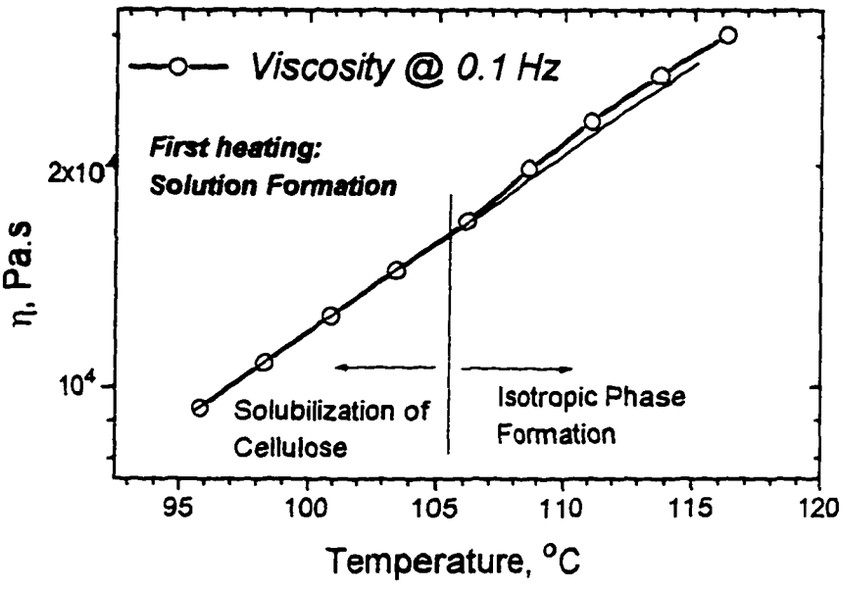


Figure 17. Dependence of viscosity on temperature for a 15% cellulosic solution of recycled CT and pulp (2:1) in NMMO·H_2O.

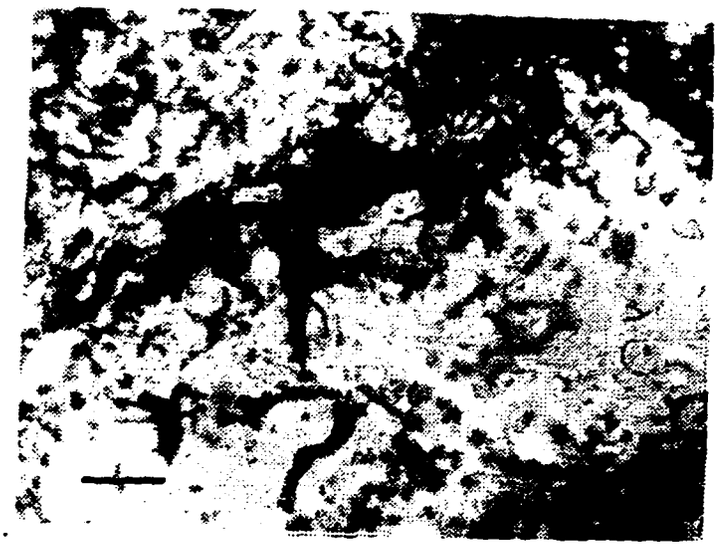


Figure 18. Microscopic liquid crystalline pattern in polarized light at 60°C of a 30% lyocell solution in NMMO·H_2O[45].

the crystallization of the solvent (the system was stable at room temperature as a supercooled liquid for hours). For a certain concentration of cellulosic solution it is advantageous to know the domain of temperature in which the system is anisotropic because: a) fibers of higher modulus are spun from mesomorphic solutions, and b) a lower η translates into a lower energy demand for spinning. Rheological measurements of a very viscous concentrated solution made with the dynamic mechanical spectrometer allowed the characterization of the transition between anisotropic and isotropic phases of cellulosic solutions. A decreasing rate of viscosity was found by plotting viscosity versus temperature as shown in Figure 19. A transition was observed between 80°C and 100°C for a 15% cellulosic solution in NMMO·H_2O as the

temperature domain in which η displayed a slow-down (η at 50 Hz) or even an increase (η at 10 Hz).

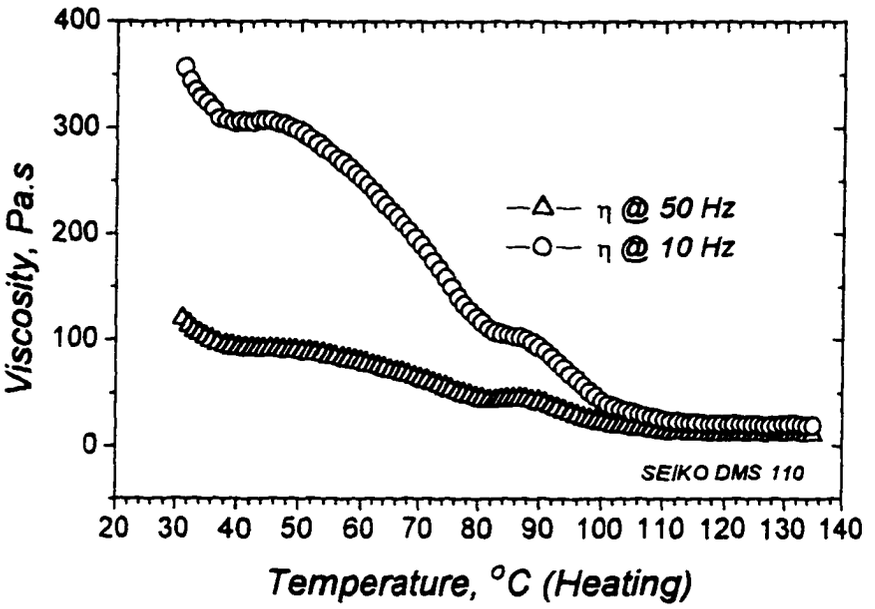


Figure 19. Dependence of viscosity on temperature for a solution of pulp (15%) in NMMO·H_2O.

The reverse phenomenon was observed in the cooling of the cellulosic solution as shown in Figure 20. As seen in this figure, the transition temperature of isotropization (determined as the temperature at which the slope of the line changes) was very much dependent upon the frequency, varying from 90°C at 0.5 Hz to 98°C at 50 Hz. This temperature domain agrees well with the transition temperatures registered during the heating of a pure pulp cellulose solution (Figure 19).

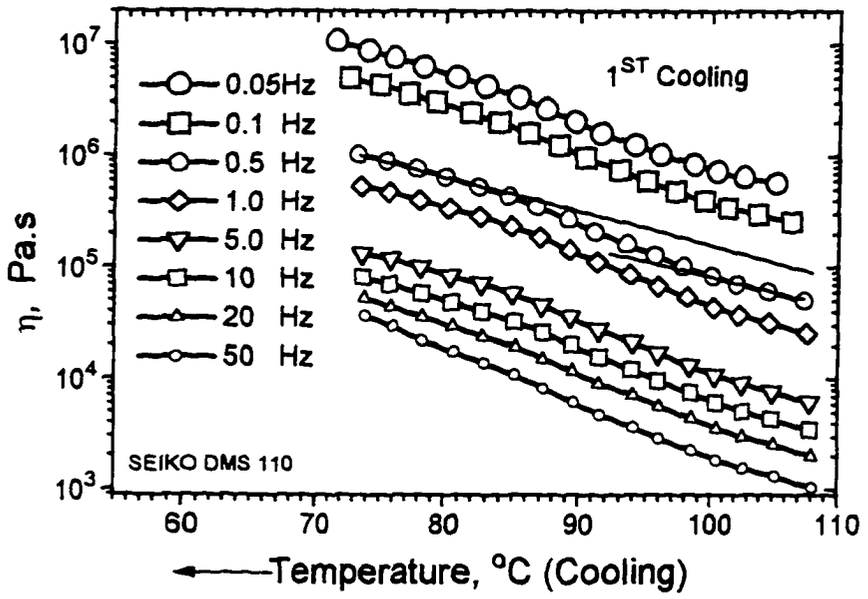


Figure 20. Rheology of 15% recycled CT:pulp (2:1) solutions in NMMO·H_2O: dependence of viscosity on temperature and frequency.

The determination of the transition temperature in the 17% solution of recovered CT in NMMO·H_2O was carried out in terms of tan δ (Figure 21). Besides frequency, this temperature was dependent, however, both on cellulose concentration and DP (the degree of polymerization of cotton cellulose is higher than that of pulp). The higher the cellulose concentration, the lower was the transition temperature which, however, increased with frequency. The maximum of tan δ was very well defined at lower frequencies (observed for example at 92°C for frequency = 0.1 Hz).

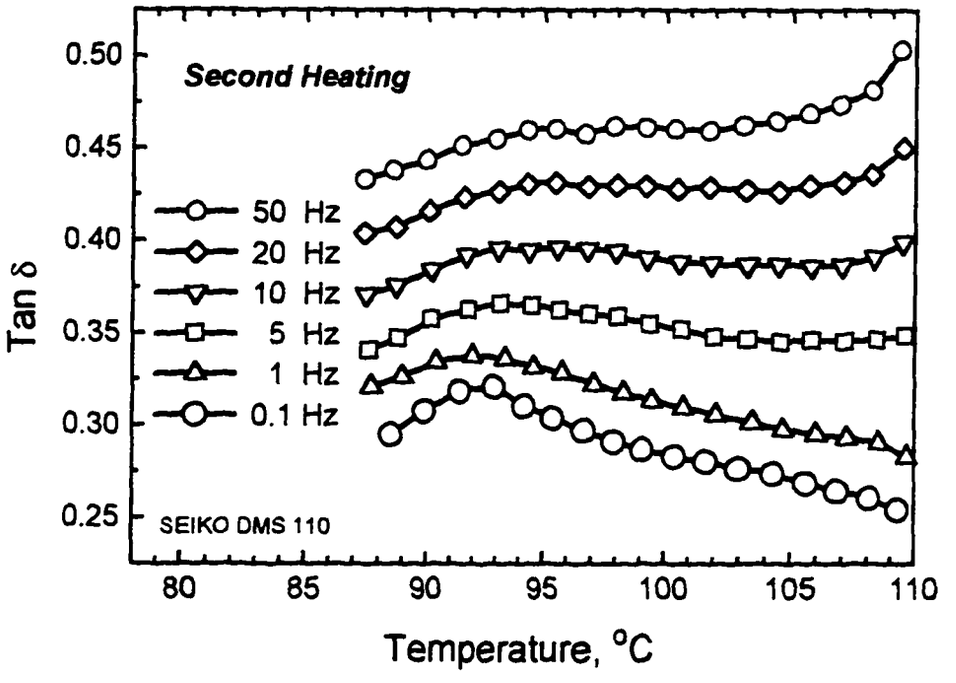


Figure 21. 17% solution of recovered CT in NMMO·H_2O: variation of tan δ with temperature and frequency.

As shown in Figure 22, the anisotropic phase in a solution of 17% recycled CT in NMMO H_2O is stable under 95°C. Microscopic observations in polarized light indicated that above this temperature the isotropization of the system occurred; in rheological measurements (η) the isotropization was indicated, as shown before, by the change of slopes. This phenomenon was reversible when the isotropic system formed at higher temperatures (> 105°C) was cooled (Figure 23).

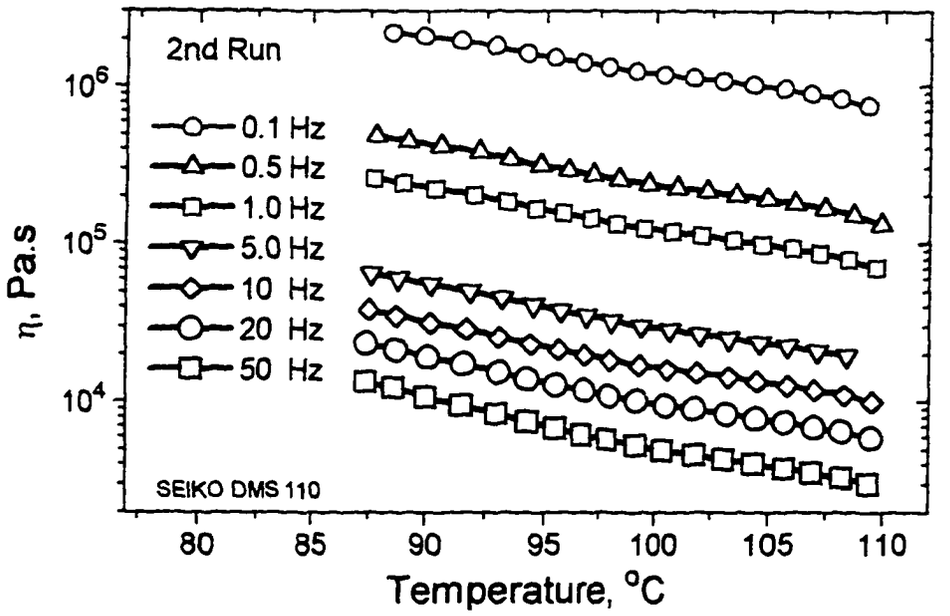


Figure 22. Variation of viscosity with temperature and frequency for a 17% cellulose solution (recycled CT) in NMMO·H_2O. 2nd heating.

Besides the previous treatments such as hydrolysis of PET in alkaline solution and subsequent bleaching in NaOCl solution, degradation of cellulose might occur in the course of solution formation from recovered CT. It is known that degradation of cellulose and solvent takes place at the same time during the dissolution of cellulose. The concentration of cellulose is another factor affecting degradation of the cellulosic solution. In general, the higher the attempted cellulose concentration, the longer the time of cellulose dissolution. Therefore, the degradation might be different depending upon the solution concentration. Cellulose solutions of recovered CT in NMMO·H_2O were prepared at the high concentrations of 12% - 18% without the addition of an anti-oxidant.

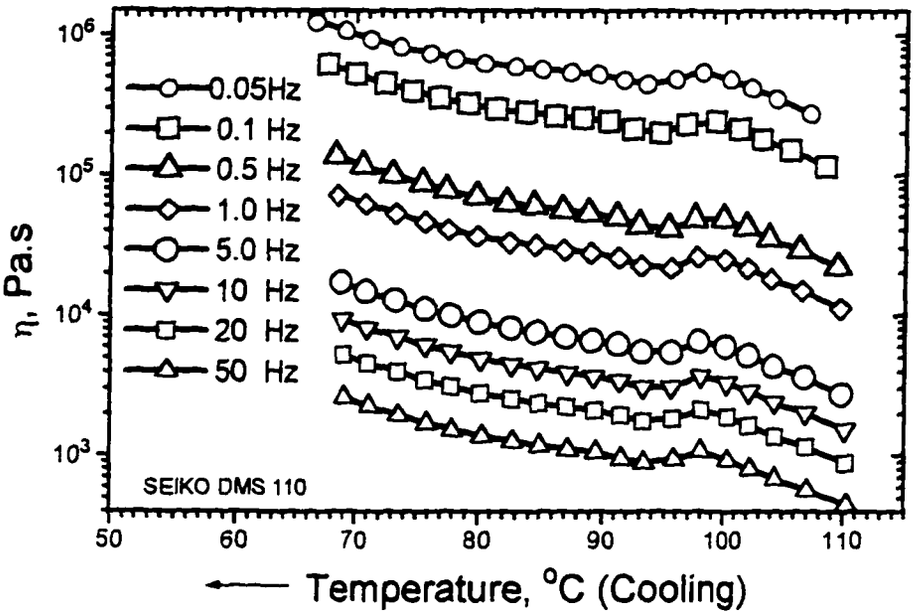


Figure 23. Variation of viscosity with decreasing temperature (cooling) and frequency for a 17% cellulose solution (recycled CT) in NMMO·H_2O.

An attempt to inhibit cellulose degradation and NMMO decomposition was made by addition of stabilizing compounds (propyl gallate). The addition of antioxidants increases the viscosity of the cellulosic solution. While a cellulose solution of 12% recovered CT was prepared readily with anti-oxidant, a cellulose concentration of 17% recovered CT resulted in poor dissolution in NMMO·H_2O. As high molecular weight cellulose from recovered CT formed lyocell solutions with high viscosity, these solutions might require high spinning temperatures which are higher than the anisotropic-isotropic transition temperature and for which the degradation could be enhanced.

The spinning of lyocell fibers from the NMMO·H_2O system at a cellulose concentration of 15% (recovered CT and pulp in a ratio of 2:1) was accomplished with the ACER instrument having the barrel and the capillary at 85°C. Fibers were spun using different strain rates. High pressures built in the rheometer barrel at low strain rates. Therefore, higher strain rates were preferred for spinning in order to avoid a severe drop of pressure at the exit from the capillaries, which might have affected drastically the geometry and properties of fibers. The diameter of the fibers spun with the ACER instrument depended also on the strain rate. Thinner and longer fibers were formed when higher strain rates were applied.

4.5. Dyeing of lyocell fibers

Before dyeing, fibers spun from the recovered CT were bleached to remove solvent color and other impurities that might inhibit uniform coloration. The fibers were bleached with 2% NaOCl solution for 10 minutes at room temperature. This short bleaching greatly improved the lightness of fibers (Figure 24). The darker color of the control fibers was attributed to the frequency of rinsing with hot water after the fiber spinning process. The shear rate during spinning in the ACER instrument seemed to affect the resultant fiber color as well as the size of fibers: a lower shear rate resulted in thicker fibers that were lighter after bleaching. In the dry state the fibers were brittle, but in wet state they were swollen and became flexible, strong and easier to handle.

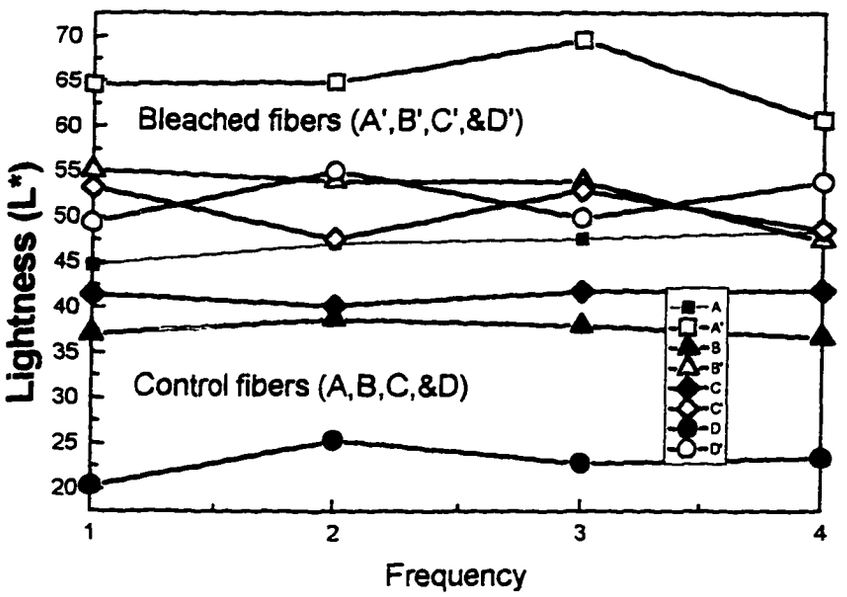


Figure 24. Lightness of fibers before and after bleaching at 2% NaOCl solution.

There are two main structural and chemical features of fibers governing their dyeability. One is permeability, the ease with which dye molecules diffuse into the fiber matrix, and the other is the presence of reactive functional groups in the molecular chains of the fiber. Three stages that govern the dyeing of textile fibers are transportation of dye through the bulk solution to the surface of the fiber, adsorption of dye at the surface of the fiber, and penetration of dye through the accessible regions of the fiber. The last step particularly is greatly dependent on the physical structure of the fiber.

As the lyocell fibers from the recovered CT are cellulosic, the reactive functional group dictating their dyeability is the same as that of all cellulosic fibers, such as CT, R, or polynosic fibers. Differences in the dyeability of these

fibers depend on their structural morphology. The lyocell fibers spun from the recovered CT in the study should be similar in dyeability to the commercial lyocell fiber, Tencel.

To predict the dyeability of lyocell fibers from recovered CT, the degree of water imbibition of a fiber (a measure of the water holding capacity of a textile fiber) was measured to determine the permeability of the fiber in dyeing. The water imbibition of R, CT, and polynosic fibers, is 90% and 50% and 75% respectively. The difference in water absorptivity between R and CT is due to differences in the orientation and crystallinity of those fibers. The crystallinity of R is 40%, while that of CT is 85%, and that of high wet modulus polynosic fibers is 50-55%. The crystallinity of Tencel is believed to be even higher than polynosic fibers because its tenacity is higher. Since an increase in crystallinity increases the glass transition temperature (Tg) of the polymer, the mobility of the dye molecule decreases in the molecular segments of the high crystalline fiber. The water imbibition of the lyocell fibers spun from the recovered CT without a drawing process was 85%, which implied that the dyeability of the lyocell fiber might be similar to that of R especially in considering the fiber morphology.

In addition DP of cellulosic chains in the those fibers is another factor that might affect the dyeability of those fibers. Due to its low DP (250), R has smaller crystals while CT (with a DP of 3000) has fibrils and microfibrils arranged in neatly ordered layers and more or less aligned with the axis of the fiber. Dyes can penetrate the R fibers more easily resulting in higher color

strength. With possible degradation of DP in the process of CT/PET recycling, due to hydrolysis of PET, bleaching of CT residue, and/or bleaching of spun recovered fibers prior to dyeing, the DP of recovered lyocell fibers should be higher than R or polynosic fibers, and lower than CT. It has been reported that dyeing with reactive dyes the dyeability of Tencel was closer to that of R than to CT [9]. However, dyeing lyocell fabric with direct dyes in another study more closely resembled that of CT than R [10]. Those results may be related to the DP and crystallinity of lyocell fibers. Wood pulp as a raw material for the lyocell fabric was closely related to the similar dyeability to the reactive dyes while the fiber morphology with high crystalline region of lyocell fabric was related to the similar dyeability to the direct dyes. The fibers spun from recovered CT in the study might show similar dyeability to that of Tencel, but the absence of drawing process and assumed higher DP might result in better dyeing with both direct and reactive dyes.

The direct dye Red 81 which has a low molecular weight was used for dyeing the lyocell fiber spun from the 15% cellulose solution (a mixture of recovered CT and pulp at the ratio of 2:1). The low molecular weight direct dye showed good penetration because of its small size as direct dyes reacted with cellulose fibers in more physical diffusion as well as in weak chemical bonding formation. Figures 25 and 27 show evidence of relatively deep dye penetration from the surface of fiber, which indicates an amorphous region formed around surface due to no application of drawing operation in the fiber production procedure. The core portion of the fiber was not dyed, implying the larger

crystalline region further evidence of crystals is seen when the fiber was viewed under crossed polars. Figures 26-27 also show the longitudinal appearance of a well dyed lyocell fiber in the microscopic test.

In dyeing of the lyocell fiber with a reactive dye the core portion of the fiber was not well dyed, further confirmation of a crystalline region in the center of the fiber. Even though a good result of dyeing with a reactive dye was shown on the surface of the fiber (Figures 28 and 30), the high molecular weight reactive dye not penetrates deep under the surface of the fiber. The reactive dye did not form a strong chemical bonds with the fiber for a good wash fastness, being only on the surface of the fiber. The loss of color was observed as a dyed fiber was left in the water for a day. The main reason for the poor dyeing result with a reactive dye might be the dyeing procedure. It did not include an increase in temperature for good exhaustion of the dye and for enhancement of cellulose fiber swelling. Figure 29 shows the longitudinal appearance of a dyed lyocell fiber. Figures 26 and 29 also show the irregular diameter of a fiber due to no drawing operation in the fiber spinning process.

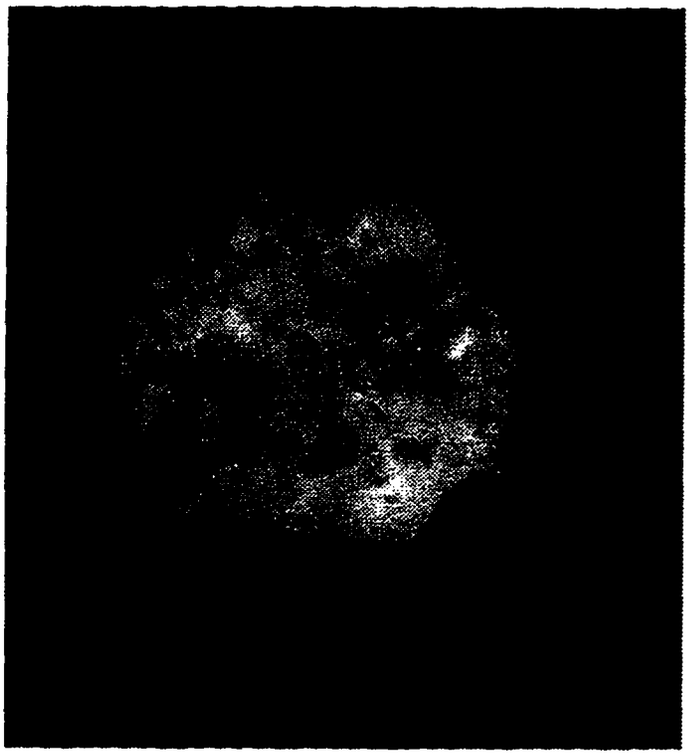


Figure 25. Cross section of a lyocell fiber dyed with a direct dye.

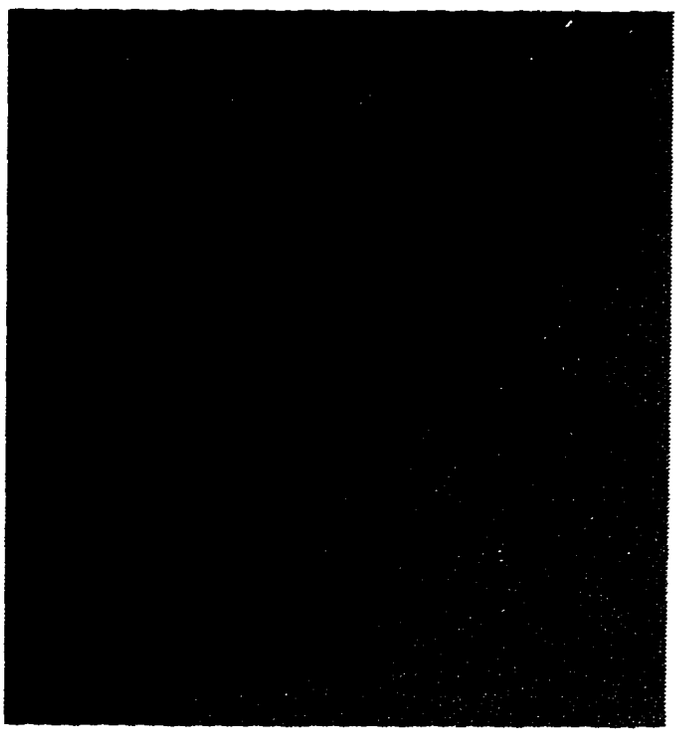


Figure 26. Longitudinal appearance of a lyocell fiber dyed with a direct dye.

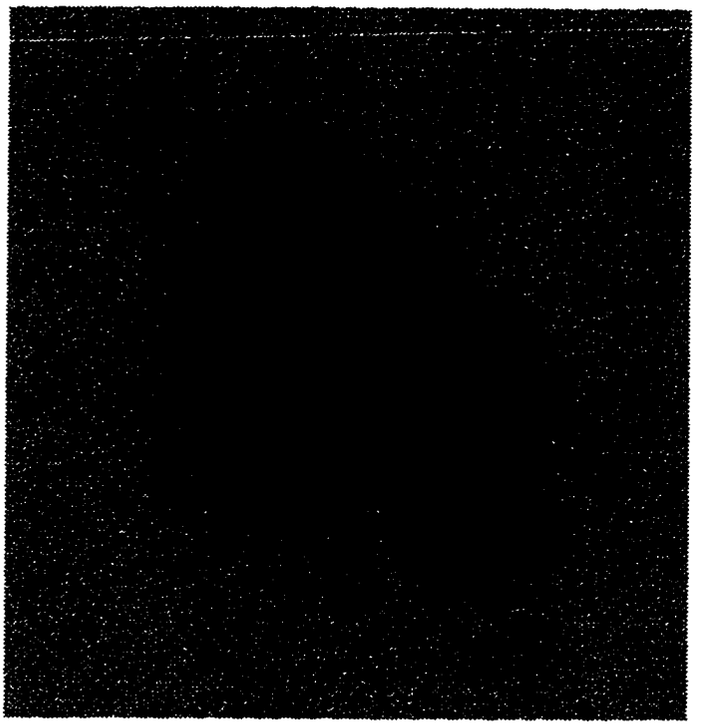


Figure 27. Lyocell fibers dyed with a direct dye.

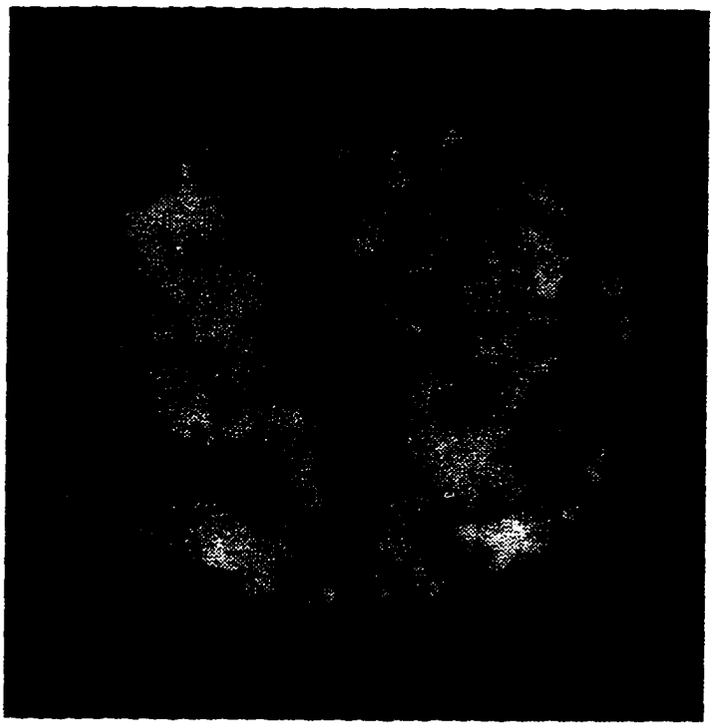


Figure 28. Cross section of a lyocell fiber dyed with a reactive dye.

Figure 29. Longitudinal appearance of a lyocell fiber dyed with a reactive dye.

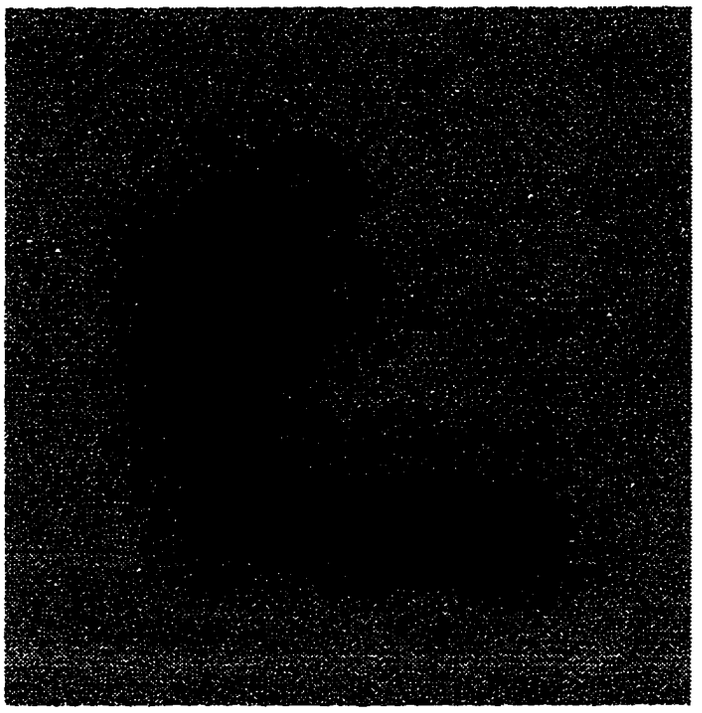


Figure 30. Lyocell fibers dyed with a reactive dye.

5. CONCLUSIONS

The thermal analytical methods, DSC and TG/DTA, were found to be efficient in determining the fiber content of CT/PET and R/N blends by providing quantitative and qualitative data. Specifically the enthalpy change, ΔH, at the melting point of PET was used for calculating the blend ratio in CT/PET blends, based on a value of ΔH (54.1 mj/mg) for 100% PET. The moisture content of the blends was also determined using TG/DTA. These thermal methods were also used to confirm complete separation of the textile components in blends.

Two separation procedures for CT/PET blends were investigated: 1) hydrolysis of PET in an aqueous NaOH solution and 2) dissolution of the CT component in an NMMO·H_2O solvent system. As the alkaline treatment of the CT/PET blends was more economical, it was selected as the primary separation technique for recycling these textile blends. After hydrolysis of the PET component, the CT residue was bleached with 2% NaOCl at room temperature. These conditions yielded the highest whiteness values with minimum degradation of the CT. Limiting the degradation of the CT residue is important in complete recycling because high molecular weight cellulose resulted in a high quality fiber in the subsequent spinning process.

The rheological properties of cellulose solutions of 15% recovered CT mixed with pulp and 17% recovered CT were determined. A liquid crystalline

phase developed in these solutions on cooling or reheating, as indicated by the peaks in tan δ at temperatures below 100°C signifying an increase in viscosity. The information on the transition between anisotropic and isotropic phases of the cellulose solutions was valuable in investigating optimum conditions of fiber extrusion of lyocell fibers from the recovered CT. Lyocell fibers were successfully spun from the 15% CT and pulp mixture (at the ratio of 2:1) using the ACER instrument, even though further study should be carried out to reduce and uniform the fiber diameter and to increase the fiber orientation. The surfaces of spun fibers from recovered CT were uniformly dyed with a direct dye and a reactive dye even though the core portion of the fibers were not dyed due to high crystallinity. Further study should be focused on developing the technique to reduce the fiber diameter in the spinning process and investigating the possible market for the recycled fiber and products.

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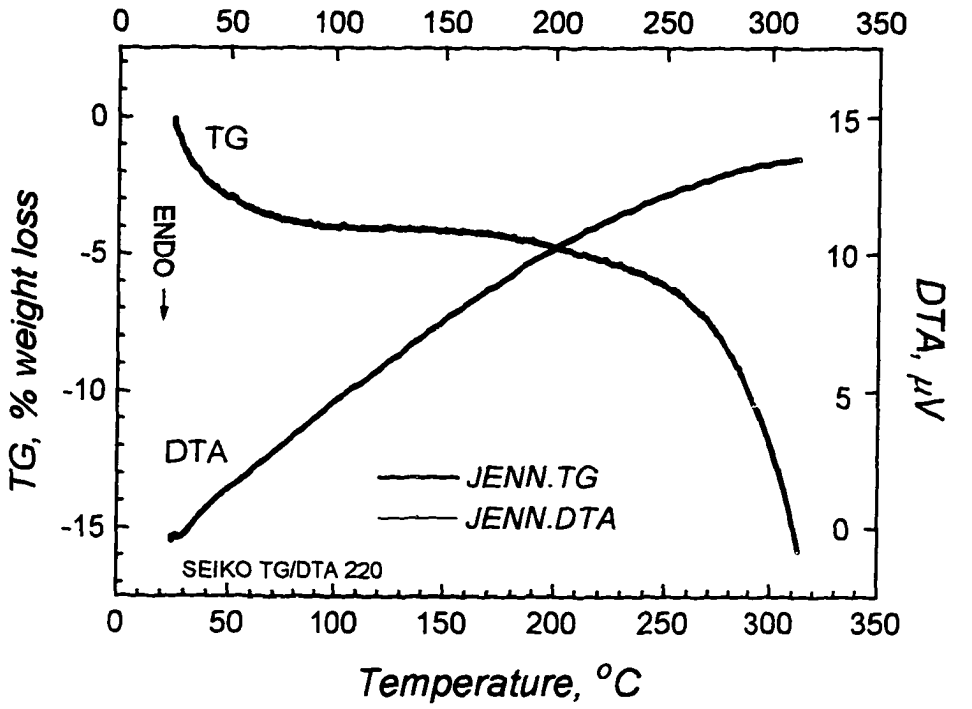
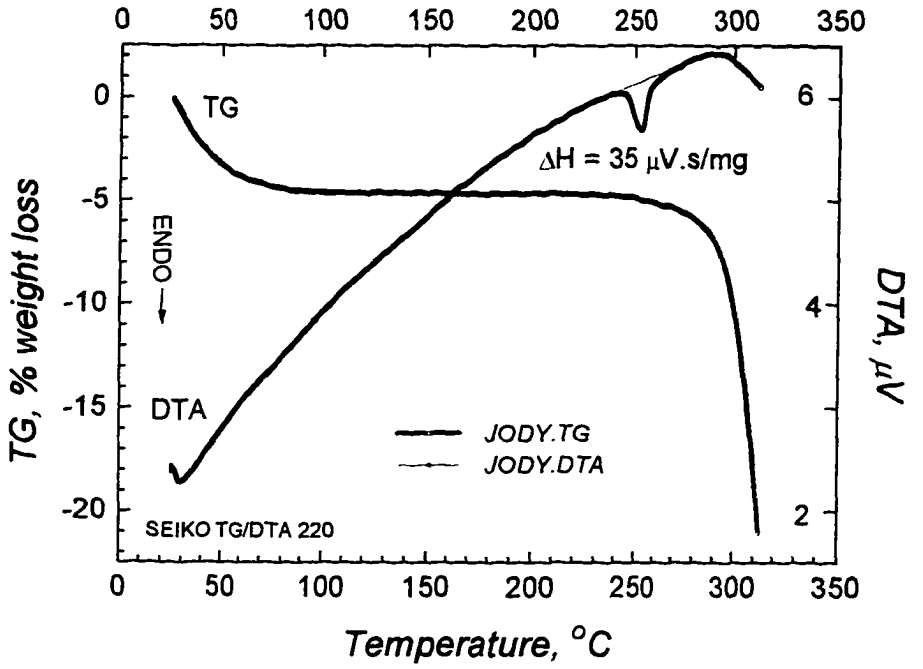
APPENDIXES

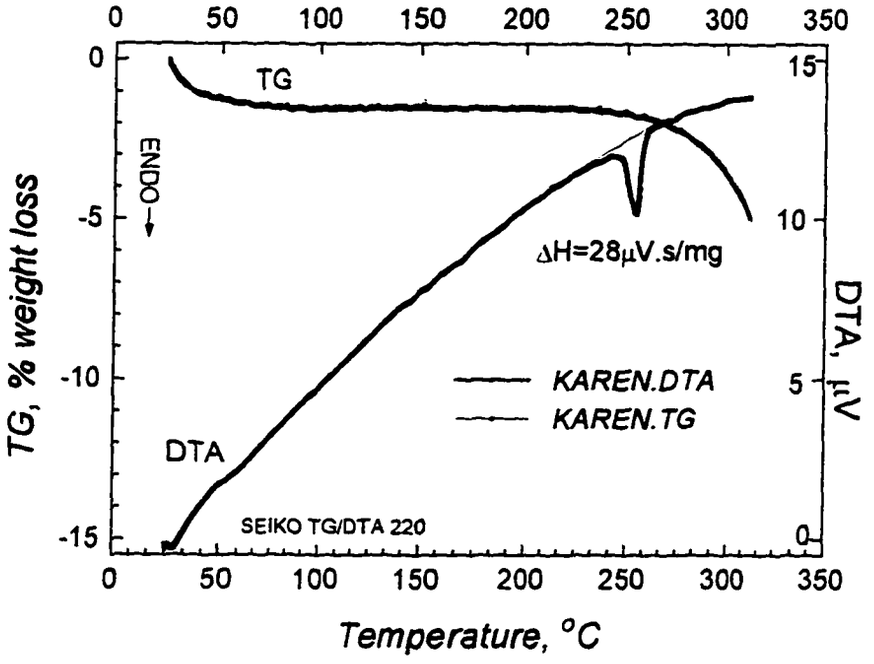
APPENDIX 1: THERMOGRAMS OF COMMERCIAL PRODUCTS.



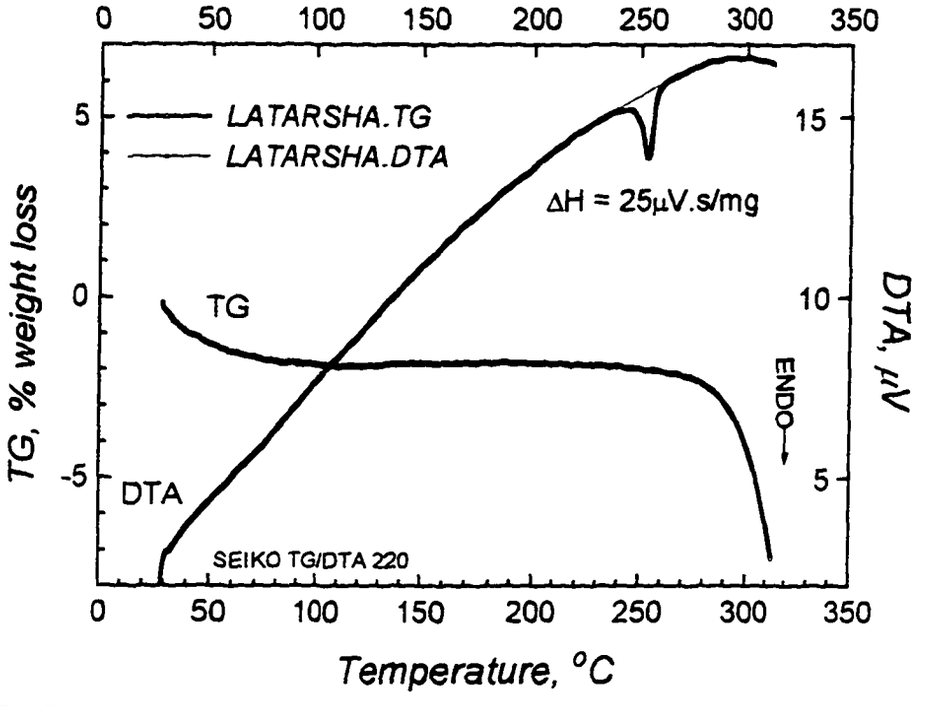
1-1. TG/DTA thermogram of *Jennifer* fabric (100% CT). Found: 100% CT.



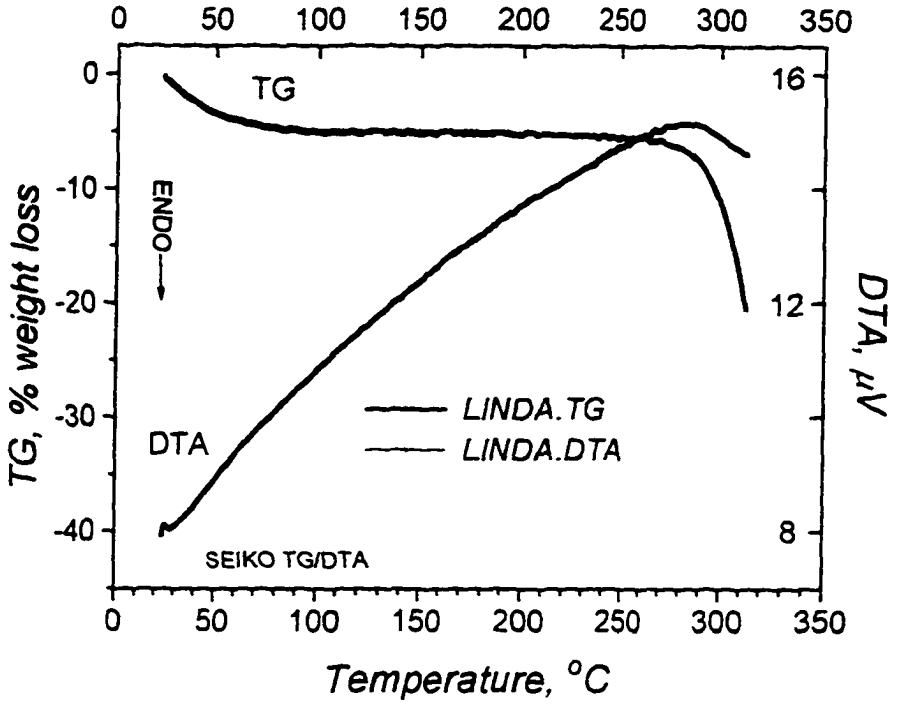
1-2. TG/DTA thermograms of *Jody* fabric (50% CT and 50% PET). Found: 65% PET.



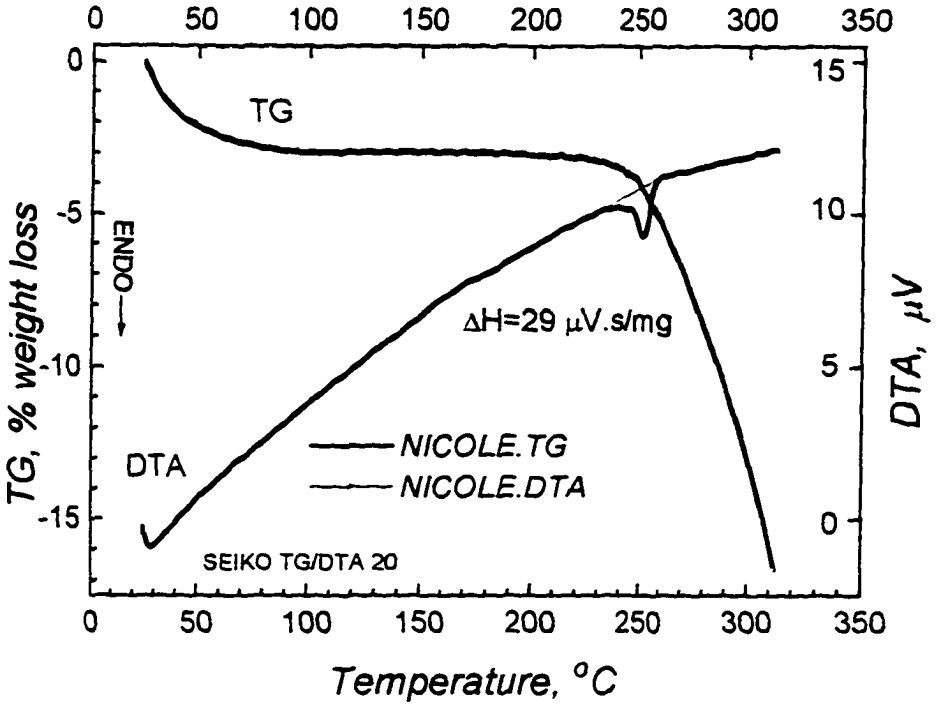
1-3. TG/DTA thermograms of *Karen* fabric (65% CT and 35% PET). Found: 52% PET.



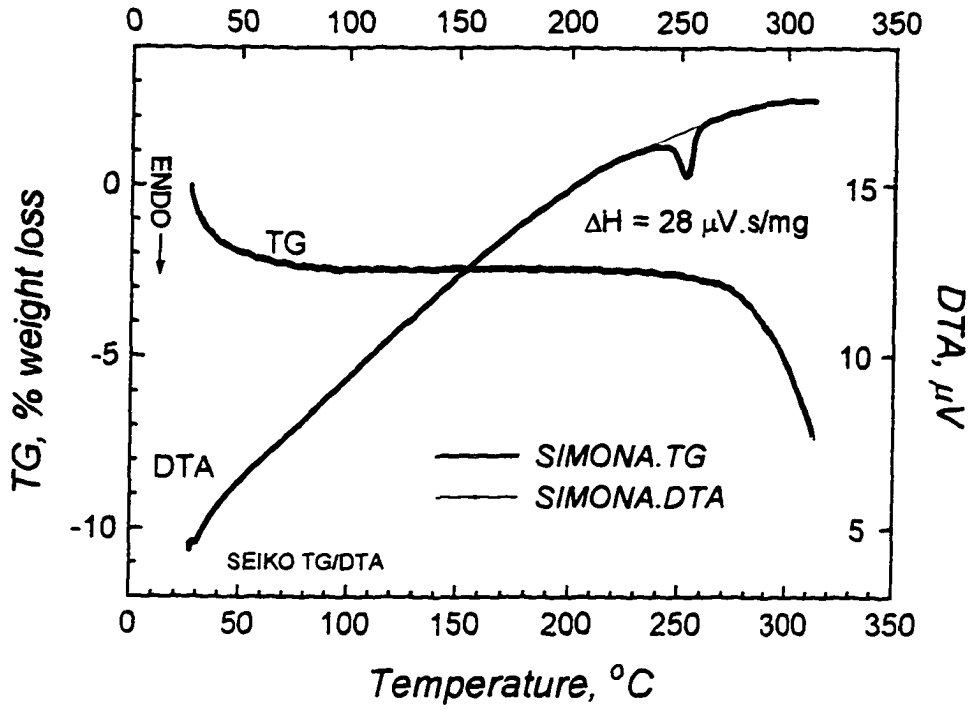
1-4. TG/DTA thermograms of *LaTarsha* fabric (100% CT). Found: 46% PET.



1-5. TG/DTA thermograms of *Linda* fabric (100% CT). Found: 100% CT.

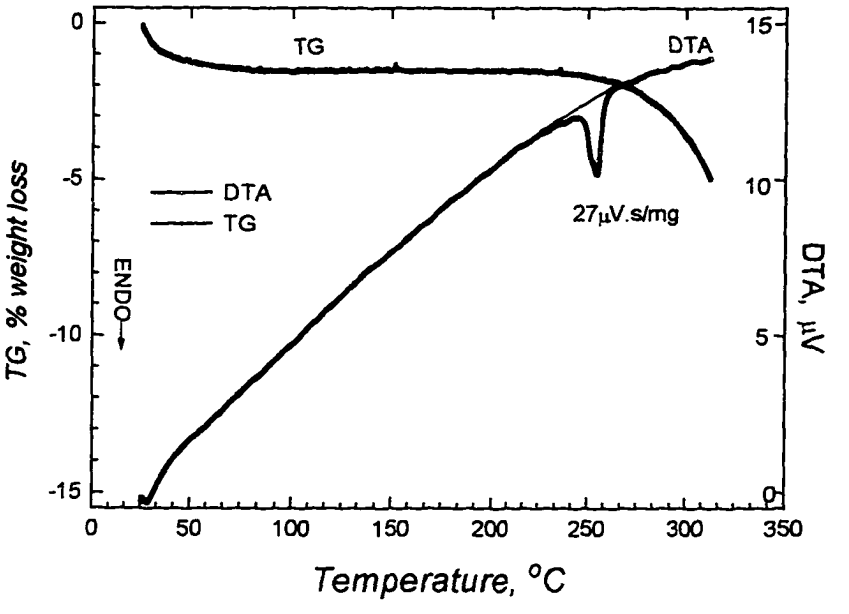


1-6. TG/DTA Thermograms of *Nicole* fabric (30% R and 70% PET). Found: 54% PET.

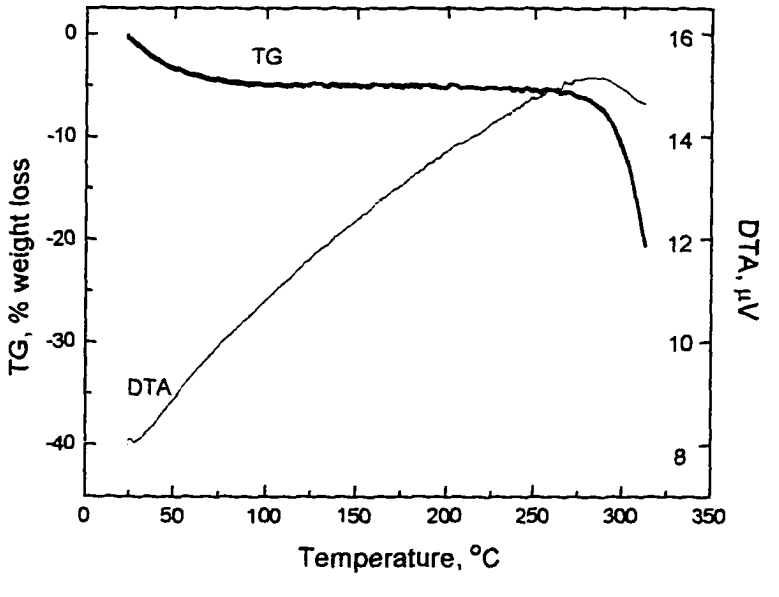


1-7. TG/DTA thermograms of *Simona* fabric (100% CT). Found: 52% PET.

APPENDIX 2: CONFIRMATION FOR COMPLETE HYDROLYSIS OF PET COMPONENT IN 50%/50% CT/PET BLACK BLEND WITH A THERMAL ANALYTICAL METHOD BEFORE (A) AND AFTER (B) ALKALINE TREATMENT.



(a)



(b)

APPENDIX 3: RESULTS OF STATISTICAL ANALYSIS FOR THE BLEACHED FABRICS.

Lightness (L*)

Fiber content	color	F-value	Pr>F	R-square
100% CT	pink	112.20	0.0001	0.970348
100% CT	navy	20.65	0.0001	0.857609
50%/50% PET/CT	pink	6.25	0.0084	0.609790
50%/50% PET/CT	black	5.90	0.0103	0.595965
50%/50% PET/CT	various	1.34	0.2756	0.280856*
65%/35% PET/CT	purple	33.89	0.0001	0.908129
65%/35% PET/CT	blue	446.28	0.0001	0.992376

Breaking strength (Warp)

Fiber content	color	F-value	Pr>F	R-square
100% CT	pink	12.66	0.0001	0.847115
100% CT	navy	48.64	0.0001	0.955117
50%/50% PET/CT	pink	0.92	0.4742	0.256289
50%/50% PET/CT	black	0.06	0.9783	0.022784
50%/50% PET/CT	various	12.44	0.0001	0.844731*
65%/35% PET/CT	purple	7.28	0.0005	0.760964
65%/35% PET/CT	blue	62.76	0.0001	0.964860

Breaking strength (Filling)

Fiber content	color	F-value	Pr>F	R-square
100% CT	pink	0.85	0.5627	0.271422
100% CT	navy	1.81	0.1540	0.442027
50%/50% PET/CT	pink	2.73	0.1137	0.506006
50%/50% PET/CT	black	5.25	0.0271	0.663132
50%/50% PET/CT	various	31.57	0.0001	0.932493*
65%/35% PET/CT	purple	5.04	0.0035	0.688151
65%/35% PET/CT	blue	6.59	0.0009	0.742552

* a printed fabric

APPENDIX 4: RESULTS OF STATISTICAL ANALYSIS FOR THE ALKALINE TREATED AND BLEACHED FABRICS.

Lightness (L*)

Fiber content	color	F-value	Pr>F	R-square
100% CT	red	1270.39	0.0001	0.997057
50%/50% PET/CT	pink	56.7	0.0001	0.938010
50%/50% PET/CT	black	895.16	0.0001	0.995828
50%/50% PET/CT	various	239.31	0.0001	0.984572*
65%/35% PET/CT	blue	1451.80	0.0001	0.997766
65%/35% PET/CT	purple	120.08	0.0001	0.967718

Breaking strength (Warp)

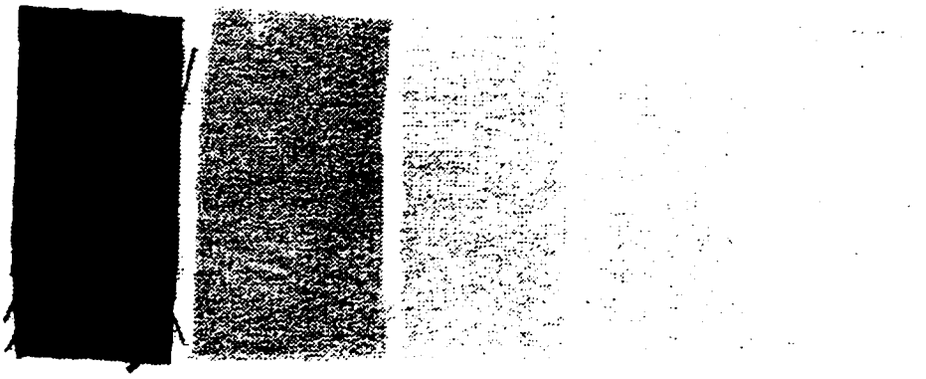
Fiber content	color	F-value	Pr>F	R-square
100% CT	red	30.30	0.0001	0.923770
50%/50% PET/CT	pink	112.52	0.0001	0.978265
50%/50% PET/CT	black	1032.70	0.0001	0.997585
50%/50% PET/CT	various	284.13	0.0001	0.991278*
65%/35% PET/CT	blue	664.68	0.0001	0.996253
65%/35% PET/CT	purple	6190.09	0.0001	0.999596

Breaking strength (Filling)

Fiber content	color	F-value	Pr>F	R-square
100% CT	red	61.29	0.0001	0.960806
50%/50% PET/CT	pink	228.82	0.0001	0.989192
50%/50% PET/CT	black	57.95	0.0001	0.956008
50%/50% PET/CT	various	255.19	0.0001	0.990298*
65%/35% PET/CT	blue	1132.01	0.0001	0.997796
65%/35% PET/CT	purple	5557.22	0.0001	0.995534

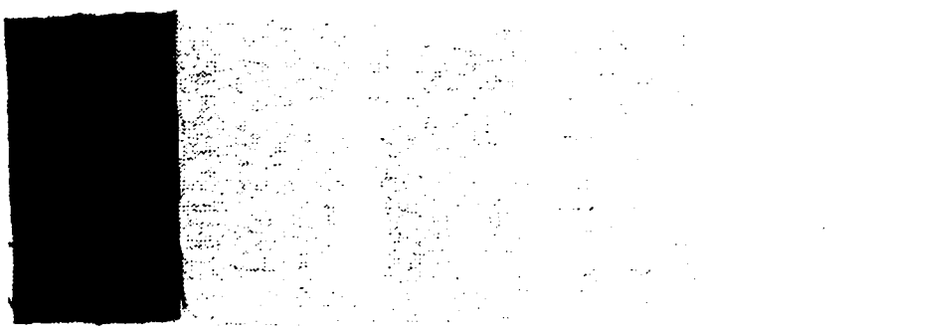
* a printed fabric

APPENDIX 5: EXAMPLES FOR BLEACHED ONLY, AND ALKALINE TREATED AND BLEACHED FABRICS.



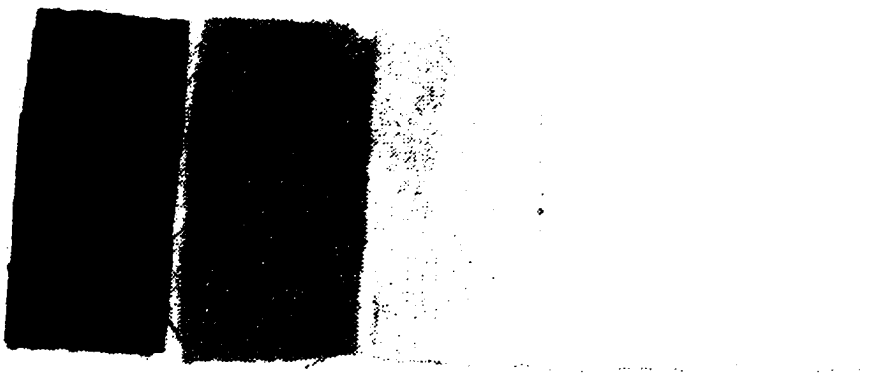
A B C D E

A: Control: 100% CT
B: After alkaline treatment
C: Bleached CT for 0.5hr
D: Bleached CT for 1hr
E: Bleached CT for 1.5hrs



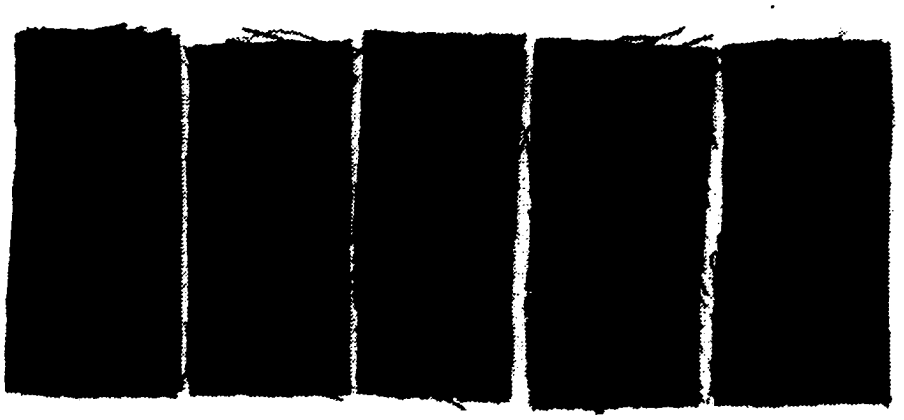
A' B' C' D' E'

A': Control: 100% CT
B': Bleached CT for 0.5hr
C': Bleached CT for 1hr
D': Bleached CT for 1.5hrs
E': Bleached CT for 2hrs



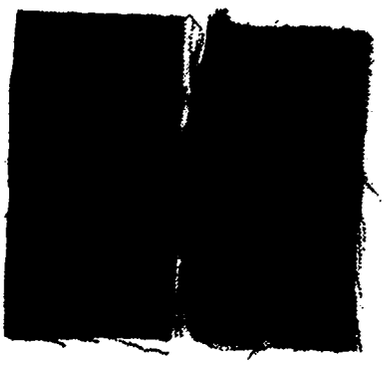
A B C D E

A: Control: 50% CT/50% PET
B: After hydrolysis of PET
C: Bleached CT for 0.5hr
D: Bleached CT for 1hr
E: Bleached CT for 1.5hrs



A' B' C' D' E'

A': Control: 50% CT/50% PET
B': Bleached CT/PET for 0.5hr
C': Bleached CT/PET for 1hr
D': Bleached CT/PET for 1.5hrs
E': Bleached CT/PET for 2hrs



A B C D E

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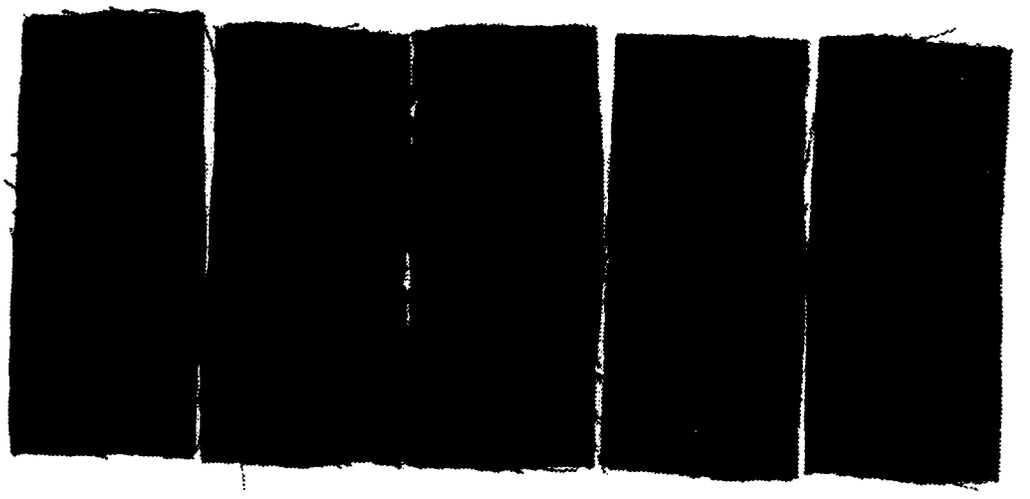
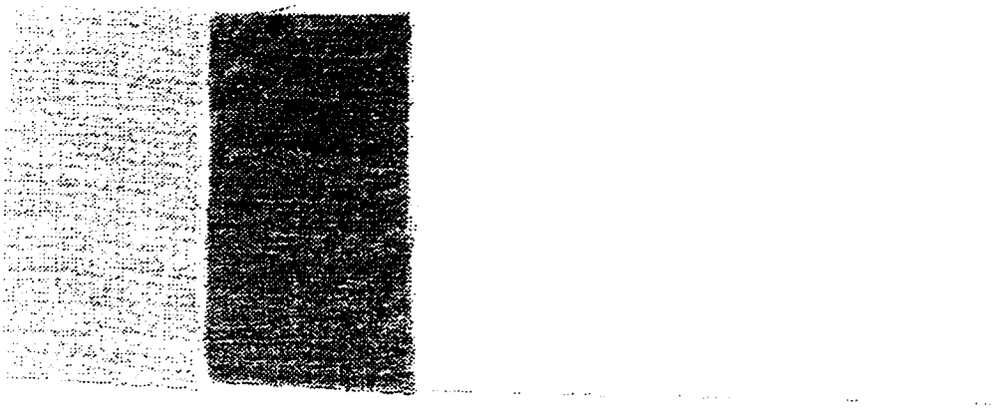
D: Bleached CT for 1hr
E: Bleached CT for 1.5hrs



A' B' C' D' E'

A': Control: 35% CT/65% PET
B': Bleached CT/PET for 0.5hr
C': Bleached CT/PET for 1hr
D': Bleached CT/PET for 1.5hrs
E': Bleached CT/PET for 2hrs



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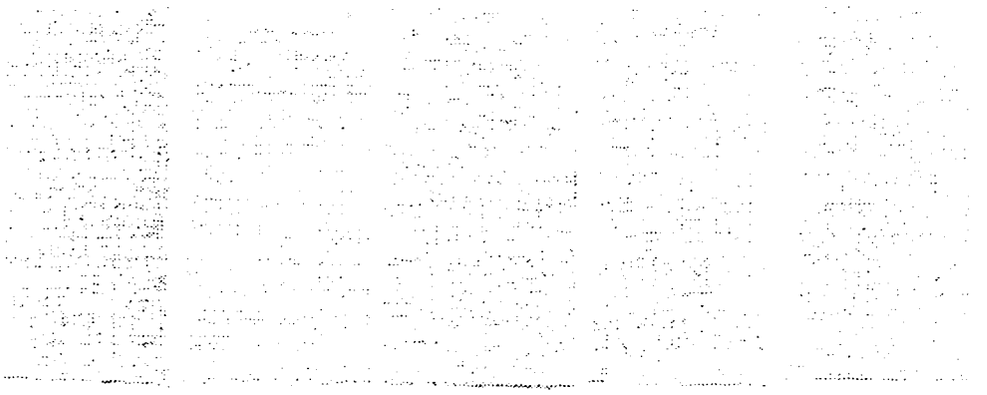
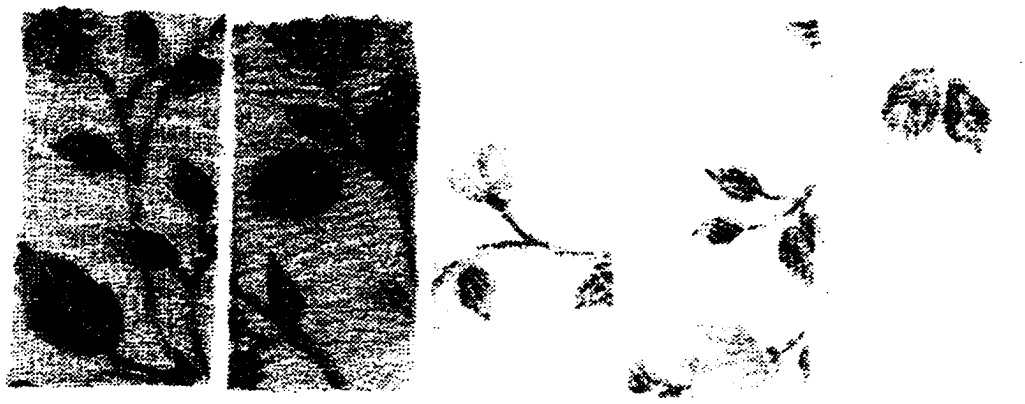
D: Bleached CT for 1hr
E: Bleached CT for 1.5hrs



A' B' C' D' E'

A': Control: 50% CT/50% PET
B': Bleached CT/PET for 0.5hr
C': Bleached CT/PET for 1hr
D': Bleached CT/PET for 1.5hrs
E': Bleached CT/PET for 2hrs

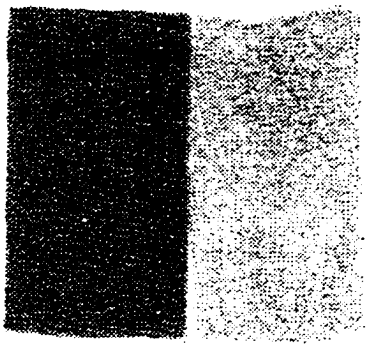


A B C D E

A: Control: 50% CT/50% PET
B: After hydrolysis of PET
C: Bleached CT for 0.5hr
D: Bleached CT for 1hr
E: Bleached CT for 1.5hrs

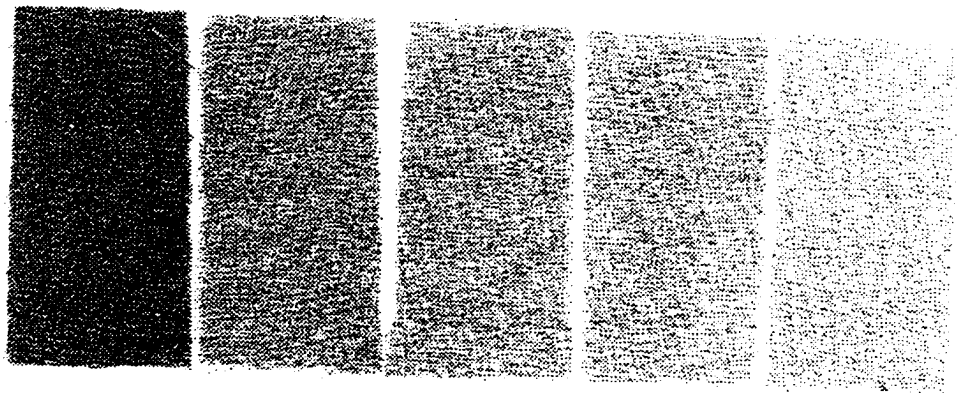
A' B' C' D' E'

A': Control: 50% CT/50% PET
B': Bleached CT/PET for 0.5hr
C': Bleached CT/PET for 1hr
D': Bleached CT/PET for 1.5hrs
E': Bleached CT/PET for 2hrs



A B C D E

A: Control: 35% CT/65% PET
B: After hydrolysis of PET
C: Bleached CT for 0.5hr
D: Bleached CT for 1hr
E: Bleached CT for 1.5hrs



A' B' C' D' E'

A': Control: 50% CT/50% PET
B': Bleached CT/PET for 0.5hr
C': Bleached CT/PET for 1hr
D': Bleached CT/PET for 1.5hrs
E': Bleached CT/PET for 2hrs

APPENDIX 6: RAW DATA

6-1. Hydrolysis of PET in alkaline treatment

1) Temp.: 103-108°C

No.	NaOH (M)	PET (g)	CT (g)	EG (ml)	Time (min)	CT (L"-W")	CT-St (lb)
10	1 (4%)	0.15	0.40	0.15	280	5.56-0.88	51.44
6	1.5 (6%)	0.15	0.40	0.15	240	5.75-0.94	40.54
7	2 (8%)	0.14	0.41	0.14	110	4.94-0.94	45.18
32	2.5 (10%)	0.14	0.40	0.14	102	5.13-0.94	44.80
1	3 (12%)	0.14	0.40	0.14	70	4.94-0.88	40.08
12	4 (16%)	0.14	0.40	0.14	120	5.00-0.88	51.06
14	5 (20%)	0.14	0.41	0.14	85	5.00-0.88	43.09

2) Temp.: 103-108°C

No.	NaOH (M)	PET (g)	CT (g)	EG (ml)	Time (min)	CT (L"-W")	CT-St (lb)
11	1 (4%)	0.29	0.41	0.29	280	5.06-0.88	49.50
5	1.5 (6%)	0.27	0.39	0.27	240	5.75-0.94	40.97
8	2 (8%)	0.29	0.39	0.29	130	5.00-0.88	45.18
30	2.5 (10%)	0.28	0.40	0.28	102	5.00-0.88	42.17
3	3 (12%)	0.28	0.39	0.28	80	4.94-0.88	40.08
13	4 (16%)	0.29	0.40	0.29	140	5.44-0.94	46.90
15	5 (20%)	0.29	0.41	0.29	70	4.94-0.88	53.21

3) Temp.: 95-100°C

No.	NaOH (M)	PET (g)	CT (g)	EG (ml)	Time (min)	CT (L"-W")	CT-St (lb)
19	1 (4%)	0.13	0.41	0.13	1020	5.75-0.94	33.93
16	1.5 (6%)	0.14	0.40	0.14	650	5.20-0.94	29.73
9	2 (8%)	0.15	0.40	0.15	365	5.75-0.94	46.55
33	2.5 (10%)	0.14	0.40	0.14	350	5.81-0.94	42.79
2	3 (12%)	0.13	0.41	0.13	270	5.56-0.94	37.19
17	4 (16%)	0.14	0.40	0.14	150	5.20-0.88	56.40
18	5 (20%)	0.14	0.39	0.14	160	4.94-0.88	49.18

4) Temp.: 95-100°C

No.	NaOH (M)	PET (g)	CT (g)	EG (ml)	Time (min)	CT (L"-W")	CT-St (lb)
21	1 (4%)	0.29	0.40	0.29	1650	5.75-0.94	35.49
22	1.5 (6%)	0.29	0.39	0.29	765	5.81-0.94	27.50
20	2 (8%)	0.28	0.41	0.28	370	5.69-094	49.29
31	2.5 (10%)	0.28	0.39	0.28	430	5.13-0.94	44.43
4	3 (12%)	0.27	0.41	0.27	210	5.05-0.94	47.14
23	4 (16%)	0.29	0.40	0.29	190	5.06-0.88	47.30
24	5 (20%)	0.29	0.40	0.29	170	4.44-0.88	45.58

5) Breaking strength depending on NaOH concentrations

	NaOH (different concentrations)							
	0M	data	1M	1.5M	2M	2.5M	3M	4M	5M
CT-St	49.61	1)	51.44	40.54	45.18	44.80	40.08	51.06	43.09
(lb)	48.08	2)	49.50	40.97	33.04	42.17	41.69	46.90	53.21
	50.20	3)	33.93	29.73	46.55	42.79	37.19	56.40	49.18
	47.03	4)	35.49	27.50	49.29	44.43	47.14	47.30	45.58
	50.47								47.30
									46.60
AVG	49.08		42.59	34.69	43.52	43.55	41.53	50.42	47.49
SD	1.47		9.16	7.07	7.19	1.27	4.18	4.41	3.45
CV	3.00		21.50	20.38	16.52	2.91	10.07	8.74	7.26

6-2. Bleaching of fabrics without alkaline treatment

0: control (washed)

0.5: bleached fabrics (with 2% NaOCl) for 30 mins

1: bleached fabrics (with 2% NaOCl) for 1 hr

1.5: bleached fabrics (with 2% NaOCl) for 1 hr and 30 mins

2: bleached fabrics (with 2% NaOCl) for 2 hrs

2.5: bleached fabrics (with 2% NaOCl) for 2 hrs and 30 mins

3: bleached fabrics (with 2% NaOCl) for 3 hrs

4: bleached fabrics (with 2% NaOCl) for 3 hrs and 30 mins

6-2-1. Lightness of bleached fabrics without alkaline treatment

1) Pink 100% CT

time(hr)	L*			
0	66.32	67.38	65.58	66.57
0.5	85.21	87.75	86.77	80.24
1	86.22	87.51	86.42	84.52
1.5	88.75	88.22	89.79	88.10
2	89.15	88.41	87.79	87.30
2.5	87.85	89.55	87.78	86.64
3	88.48	88.56	87.83	89.61
4	89.77	88.73	88.78	89.32

2) Navy blue 100% CT

time(hr)	L*			
0	17.61	17.71	18.07	17.71
0.5	19.48	18.69	18.95	19.48
1	19.79	19.62	19.46	20.06
1.5	19.98	19.20	21.04	20.19
2	20.57	20.54	20.96	20.22
2.5	20.75	20.25	20.02	20.65
3	22.36	21.06	20.78	20.44
4	22.72	21.60	23.82	21.52

3) Pink 50%/50% CT/PET

time(hr)	L*			
0	71.12	70.31	71.28	72.50
0.5	71.49	70.98	71.61	70.00
1	73.09	71.24	70.49	71.21
1.5	73.76	71.49	73.33	73.90
2	72.44	73.78	73.42	73.61

4) Black 50%/50% CT/PET

time(hr)	L*			
0	16.14	16.32	15.93	16.71

0.5	31.48	31.96	31.18	31.91
1	31.98	31.96	31.84	31.69
1.5	31.18	31.00	31.27	31.34
2	32.76	31.96	31.76	31.81

5) Printed 50%/50% CT/PET

time(hr)	L*			
0	67.02	64.58	66.07	68.40
0.5	65.12	67.93	63.58	75.45
1	67.05	70.28	70.06	67.44
1.5	70.65	67.67	72.90	72.49
2	72.02	69.90	69.47	65.24
2.5	71.13	70.74	70.67	70.60
3	73.19	68.82	63.24	70.52
4	71.71	70.48	71.44	71.92

6) Blue 35%/65% CT/PET

time(hr)	L*			
0	19.52	20.25	20.01	20.44
0.5	31.16	31.07	30.82	32.39
1	33.08	32.12	33.00	32.40
1.5	33.80	33.47	33.36	33.23
2	34.15	34.00	33.16	33.72
2.5	37.55	36.98	37.01	36.23
3	36.36	36.14	38.08	36.95
4	37.64	38.34	38.65	38.20

7) Purple 35%/65% CT/PET

time(hr)	L*			
0	56.31	56.96	56.37	50.95
0.5	61.85	62.23	62.30	62.59
1	62.67	60.52	62.86	62.01
1.5	64.45	62.89	64.23	62.37
2	64.80	64.83	64.66	64.68

2.5	63.47	65.74	64.86	66.14
3	65.84	67.23	67.36	66.31
4	65.06	66.47	66.52	66.80

6-2-2. Break at peak (lb) of bleached fabrics

1w) Pink 100% CT (warp)

time (hr)	break at peak (lb)		
0	60.32	52.94	56.54
0.5	52.24	49.72	45.53
1	40.48	35.06	35.74
1.5	43.25	41.15	41.56
2	40.00	38.66	43.95
2.5	31.40	31.05	33.96
3	29.06	23.82	26.85
4	21.47	19.67	20.14

1f) Pink 100% CT (Filling)

time (hr)	break at peak (lb)		
0	55.70	59.92	56.72
0.5	54.42	52.21	50.42
1	47.62	48.83	48.22
1.5	44.40	44.32	44.16
2	42.93	42.23	42.58
2.5	28.20	27.40	31.57
3	20.55	31.10	24.58
4	23.62	21.44	19.41

2w) Navy blue 100% CT (Warp)

time (hr)	break at peak (lb)		
0	60.13	66.42	55.19
0.5	61.85	53.80	63.41
1	54.09	67.65	55.22
1.5	56.94	56.43	51.38
2	47.76	43.65	52.35

2.5	45.15	45.74	53.10
3	30.93	40.83	38.68
4	34.63	36.29	35.38

2f) Navy blue 100% CT (Filling)

time (hr)	break at peak (lb)		
0	54.12	56.29	49.18
0.5	48.80	49.91	50.76
1	46.09	52.05	43.57
1.5	38.47	40.67	43.14
2	38.58	40.48	38.28
2.5	31.70	32.33	30.87
3	26.51	25.96	28.26
4	30.24	31.45	33.46

3w) Pink 50%/50% CT/PET (Warp)

time (hr)	break at peak (lb)		
0	88.64	88.48	68.89
0.5	73.96	71.89	73.00
1	68.49	72.53	71.59
1.5	70.69	71.69	71.59
2	73.45	72.24	69.29

3f) Pink 50%/50% CT/PET (Filling)

time (hr)	break at peak (lb)		
0	49.23	41.74	43.30
0.5	41.21	44.89	41.13
1	38.87	41.16	38.49
1.5	38.96	37.45	40.30
2	40.62	39.38	37.89

4w) Black 50%/50% CT/PET (Warp)

time (hr)	break at peak (lb)		
0	71.14	68.19	72.24

0.5	46.79	42.68	46.74
1	48.62	43.62	43.78
1.5	43.81	47.27	42.83
2	43.60	44.40	47.60

4f) Black 50%/50% CT/PET (Filling)

time (hr)	break at peak (lb)		
0	96.64	88.13	82.42
0.5	72.37	69.15	69.10
1	72.29	59.97	62.9
1.5	62.07	64.11	62.39
2	51.22	51.11	62.76

5w) Printed 50%/50% CT/PET (Warp)

time (hr)	break at peak (lb)		
0	59.65	58.07	55.70
0.5	52.35	48.29	55.54
1	49.88	49.56	53.07
1.5	44.51	48.70	50.90
2	51.27	46.98	52.43
2.5	50.93	43.78	49.34
3	52.40	48.43	47.17
4	44.91	46.76	45.69

5f) Printed 50%/50% CT/PET (Filling)

time (hr)	break at peak (lb)		
0	37.85	37.92	38.20
0.5	36.45	35.38	35.37
1	35.02	34.85	35.88
1.5	34.02	33.98	35.56
2	32.32	32.93	34.07
2.5	31.94	33.13	32.67
3	30.78	32.29	30.79
4	33.05	32.92	32.96

6w) Blue 35%/65% CT/PET (Warp)

time (hr)	break at peak (lb)		
0	89.07	88.62	95.88
0.5	91.92	96.32	87.57
1	91.11	86.44	85.90
1.5	84.46	82.12	79.68
2	89.77	88.75	87.95
2.5	86.60	76.27	87.33
3	81.93	85.09	87.81
4	76.59	77.79	69.97

6f) Blue 35%/65% CT/PET (Filling)

time (hr)	break at peak (lb)		
0	45.13	41.66	42.15
0.5	38.08	37.07	41.40
1	40.80	39.84	37.70
1.5	42.71	40.24	34.98
2	42.76	37.89	42.09
2.5	38.40	42.76	42.47
3	41.50	40.99	42.28
4	41.53	40.24	42.74

7w) Purple 35%/65% CT/PET (Warp)

time (hr)	break at peak (lb)		
0	81.29	82.68	82.39
0.5	76.19	80.19	80.19
1	81.45	80.13	80.79
1.5	83.33	76.83	75.70
2	79.11	73.29	86.47
2.5	79.68	80.37	86.04
3	76.48	77.58	78.42
4	80.48	80.43	82.79

7f) Purple 35%/65% CT/PET (Filling)

time (hr)	break at peak (lb)		
0	41.58	41.13	45.82
0.5	38.27	42.85	40.99
1	48.56	47.76	45.18
1.5	45.85	38.28	42.34
2	45.82	34.83	38.63
2.5	43.49	45.21	40.43
3	47.54	42.20	49.00
4	41.96	43.38	46.66

6-3. Alkaline treated and bleached fabrics

6-3-1. Lightness of alkaline treated and bleached fabrics

-1: control fabrics (washed)

0: alkaline treated CT (for 1.5 hrs at 4M NaOH)

0.5: alkaline treated CT (for 1.5 hrs at 4M NaOH) and bleached (with 2% NaOCl) for 30 mins

1: alkaline treated CT (for 1.5 hrs at 4M NaOH) and bleached (with 2% NaOCl) for 1 hr

1.5: alkaline treated CT (for 1.5 hrs at 4M NaOH) and bleached (with 2% NaOCl) for 1hr and 30 mins

1) Red 100% CT

time(hr)	L*			
-1	20.13	19.43	20.30	19.64
0	58.65	57.62	58.95	58.23
0.5	81.59	79.57	77.91	83.77
1	83.04	82.71	82.73	83.59
1.5	84.45	81.77	85.35	80.81

2) Pink 50%/50% CT/PET

time(hr)	L*			
-1	71.12	70.31	71.28	72.50
0	60.29	57.36	59.76	61.05

0.5	71.90	72.87	70.04	67.77
1	71.16	73.89	72.64	72.95
1.5	73.59	70.67	70.22	71.51

3) Black 50%/50% CT/PET

time(hr)	L*			
-1	16.14	16.32	15.93	16.71
0	48.00	50.52	48.26	48.64
0.5	80.67	80.25	74.70	75.66
1	82.81	79.42	80.86	78.96
1.5	77.17	78.99	81.02	81.32

4) Printed 50%/50% CT/PET

time(hr)	L*			
-1	67.02	64.58	66.07	68.40
0	59.27	56.79	57.44	56.07
0.5	78.18	78.46	79.61	77.62
1	77.29	77.70	77.49	78.44
1.5	78.36	79.46	77.24	80.73

5) Blue 35%/65% CT/PET

time(hr)	L*			
-1	19.52	20.25	20.01	20.44
0	22.00	20.22	21.87	20.45
0.5	72.83	74.99	75.00	74.30
1	76.54	76.60	74.82	75.03
1.5	76.39	76.46	76.37	75.76

6) Purple 35%/65% CT/PET

time(hr)	L*			
-1	56.31	56.96	56.37	50.95
0	56.77	56.07	57.71	58.26
0.5	76.23	75.41	74.75	78.74

1	75.28	80.08	76.02	80.43
1.5	76.06	74.16	77.67	75.45

6-3-2. Breaking strength of alkaline treated and bleached fabrics

1w) Red 100% CT (Warp)

time(hr)	break at peak (lb)		
-1	43.41	41.82	46.01
0	65.42	70.12	53.91
0.5	52.56	52.46	44.16
1	32.09	39.33	37.96
1.5	21.24	24.18	20.77

1f) Red 100% CT (Filling)

time(hr)	break at peak (lb)		
-1	26.95	27.88	28.61
0	55.06	57.88	59.54
0.5	35.82	41.88	45.18
1	35.94	30.43	27.26
1.5	22.11	20.35	19.16

2w) Pink 50%/50% CT/PET (Warp)

time(hr)	break at peak (lb)		
-1	88.64	88.48	68.89
0	16.23	15.27	15.66
0.5	13.69	14.28	13.85
1	11.23	10.46	10.88
1.5	7.71	9.64	8.98

2f) Pink 50%/50% CT/PET (Filling)

time(hr)	break at peak (lb)		
-1	49.23	41.74	43.30
0	12.65	13.12	12.52
0.5	9.70	10.03	9.31

1	9.07	8.31	9.47
1.5	7.98	8.0	7.93

3w) Black 50%/50% CT/PET (Warp)

time(hr)	break at peak (lb)		
-1	71.14	68.19	72.24
0	9.10	5.03	9.79
0.5	7.62	6.11	7.10
1	3.28	3.74	4.05
1.5	3.43	1.85	3.01

3f) Black 50%/50% CT/PET (Filling)

time(hr)	break at peak (lb)		
-1	96.64	86.13	82.42
0	15.53	12.71	15.29
0.5	11.15	9.13	11.38
1	6.10	6.44	7.23
1.5	3.86	4.31	3.64

4w) Printed 50%/50% CT/PET (Warp)

time(hr)	break at peak (lb)		
-1	59.65	58.07	55.70
0	12.46	15.04	17.87
0.5	12.84	16.25	16.57
1	13.96	9.37	9.39
1.5	7.42	7.51	7.23

4f) Printed 50%/50% (Filling)

time(hr)	break at peak (lb)		
-1	37.85	37.92	38.20
0	9.25	13.18	10.27
0.5	11.10	10.46	8.51
1	7.32	10.61	7.08
1.5	5.66	4.15	5.13

5w) Blue 35%/65% CT/PET (Warp)

time(hr)	break at peak (lb)		
-1	89.07	88.62	95.88
0	9.00	15.61	13.93
0.5	11.38	10.85	11.72
1	6.68	6.49	9.18
1.5	3.63	3.41	4.05

5f) Blue 35%/65% CT/PET (Filling)

time(hr)	break at peak (lb)		
-1	45.13	41.66	42.15
0	5.55	5.96	4.96
0.5	4.98	4.45	5.27
1	4.19	4.10	4.08
1.5	2.31	2.27	2.64

6w) Purple 35%/65% CT/PET (Warp)

time(hr)	break at peak (lb)		
-1	81.29	82.68	82.39
0	9.13	9.65	10.57
0.5	9.06	8.35	9.68
1	7.29	5.48	6.96
1.5	6.26	5.28	5.85

6f) Purple 35%/65% CT/PET (Filling)

time(hr)	break at peak (lb)		
-1	41.58	41.13	45.82
0	4.61	4.30	3.94
0.5	4.66	4.10	4.37
1	4.81	4.25	2.99
1.5	3.79	2.80	2.61

6-4. Mean (M) and standard deviation (sd) of bleached fabrics without (6-4-1) and with (6-4-2) alkaline treatment: Standard devidations were reported in the following colomn to M

6-4-1. M and sd of bleached fabrics without alkaline treatment

1) M and sd of lightness of bleached fabrics without alkaline treatment

time (hr)	CT(%)	100 (pink)	100 (navy)	35 (blue)	35 (purple)	50 (print)	50 (knit)	50 (pink)	50 (black)
0		66.45	17.78	20.06	55.15	66.52	20.27	71.30	16.28
		0.7	0.2	0.4	2.8	1.6	0.6	0.9	0.3
0.5		84.99	19.15	31.36	62.24	68.02	53.81	71.02	31.63
		3.3	0.4	0.7	0.3	5.3	0.9	0.7	0.4
1		86.17	19.73	32.65	62.02	68.71	59.92	71.51	31.87
		1.2	0.3	0.5	1.1	1.7	1.1	1.1	0.1
1.5		88.72	20.10	33.47	63.49	70.93	64.63	73.12	31.20
		0.8	0.8	0.2	1.0	2.4	0.6	1.1	0.1
2		88.16	20.57	33.76	64.74	69.16	66.67	73.31	32.08
		0.8	0.3	0.4	0.1	2.8	1.1	0.6	0.5
2.5		87.96	20.42	36.94	65.05	70.79	69.65		
		1.2	0.3	0.5	1.2	0.2	0.9		
3		88.62	21.16	36.88	66.69	69.94	71.37		
		0.7	0.8	0.9	0.7	4.2	1.8		
4		89.15	22.42	38.21	66.21	71.39	75.11		
		0.5	1.1	0.4	0.8	0.6	0.7		

2w) M and sd of breaking strength (warp) of bleached fabrics without alkaline treatment

time (hr)	CT(%)	100 (pink)	100 (navy)	35 (blue)	35 (purple)	50 (print)	50 (knit)	50 (pink)	50 (black)
0		56.60	60.58	91.19	82.12	57.81	14.78	82.00	70.52
		3.7	5.6	4.1	0.7	2.0	0.3	11.4	2.1
0.5		49.16	59.69	91.94	78.86	52.06	13.23	72.95	45.40
		3.4	5.2	4.4	2.3	3.6	3.4	1.0	2.4
1		37.09	58.99	87.82	80.79	50.84	12.96	70.87	45.34

	3.0	7.5	2.9	0.7	1.9	1.0	2.1	2.8
1.5	41.99	54.92	82.09	78.62	48.04	12.40	71.32	44.64
	1.1	3.1	2.4	4.1	3.2	1.6	0.6	2.3
2	40.87	47.92	88.82	79.62	50.23	11.24	71.66	45.20
	2.8	4.4	0.9	6.6	2.9	1.0	2.1	2.1
2.5	32.14	48.00	83.40	82.03	48.02	9.33		
	1.6	4.4	6.2	3.5	3.8	1.0		
3	26.58	36.81	84.94	77.49	49.33	8.65		
	2.6	5.2	2.9	1.0	2.7	0.8		
4	20.43	35.43	74.78	81.23	45.79	7.63		
	0.9	0.8	4.2	1.4	0.9	1.7		

2f) M and sd of breaking strength (filling) of bleached fabrics without alkaline treatment

time	CT(%)	100	100	35	35	50	50	50	50
(hr)		(pink)	(navy)	(blue)	(purple)	(print)	(knit)	(pink)	(black)
0		57.45	53.20	42.98	42.84	37.99	33.22	44.76	89.06
		2.2	3.6	1.9	2.6	0.2	5.7	4.0	7.1
0.5		52.35	49.82	38.85	40.70	35.73	29.69	42.41	70.21
		2.0	1.0	2.3	2.3	0.6	2.3	2.1	1.9
1		48.22	47.24	39.45	47.17	35.25	27.50	40.68	65.05
		0.6	4.4	1.6	1.8	0.6	3.9	1.6	6.4
1.5		44.29	40.76	39.31	42.16	34.52	21.01	38.97	62.86
		0.1	2.3	3.9	3.8	0.9	1.7	1.3	1.1
2		42.58	39.11	40.91	39.76	33.11	25.44	39.82	55.03
		0.4	1.2	2.6	5.6	0.9	1.4	0.7	6.7
2.5		29.06	31.63	41.21	43.04	32.58	22.78		
		2.2	0.7	2.4	2.4	0.6	0.6		
3		25.41	26.91	41.59	46.25	31.29	18.88		
		5.3	1.2	0.6	3.6	0.9	1.7		
4		21.49	31.72	41.50	44.00	32.98	16.72		
		2.1	1.6	1.3	2.4	0.1	0.5		

6-4-2. M and sd of bleached fabrics with alkaline treatment

1w) M and sd of lightness of bleached fabrics with alkaline treatment

time (hr)	CT(%)	100 red	35 blue	35 purple	50 pink	50 black	50 print	50 knit
-1(control)		19.88	19.77	55.15	71.30	16.28	66.52	20.27
		0.4	0.3	2.8	0.9	0.3	1.6	0.6
0(after hydrolysis)		59.62	21.14	57.20	58.36	48.85	57.39	59.73
		0.6	0.9	1.0	1.6	1.1	1.4	1.4
0.5		70.90	74.28	76.28	80.71	77.82	78.47	87.43
		2.5	1.0	1.7	1.8	3.1	0.8	0.2
1		72.66	74.25	77.95	83.01	80.51	77.73	86.68
		0.4	2.6	2.7	1.1	1.7	0.5	0.8
1.5		71.50	76.25	75.84	83.10	79.63	78.95	89.24
		2.2	0.3	1.5	1.5	1.9	1.5	0.6

2w) M and sd of breaking strength (warp) of bleached fabrics with alkaline treatment

time (hr)	CT(%)	100 red	35 blue	35 purple	50 pink	50 black	50 print	50 knit
-1(control)		43.75	91.19	82.12	82.00	70.52	57.81	14.77
		2.1	4.0	0.7	11.4	2.1	2.0	0.3
0(after hydrolysis)		63.15	12.85	9.78	15.72	7.97	15.12	31.37
		8.3	3.4	0.7	0.5	2.6	2.7	1.4
0.5		49.73	11.32	9.03	13.94	6.94	15.22	30.94
		4.8	0.4	0.7	0.3	0.8	2.1	3.8
1		36.46	7.45	6.58	10.85	3.69	10.91	18.71
		3.8	1.5	1.0	0.4	0.4	2.6	1.7
1.5		22.06	3.70	5.80	8.78	2.76	7.39	9.10
		1.8	0.3	0.5	1.0	0.8	0.1	1.4

2f) M and sd of breaking strength (filling) of bleached fabrics with alkaline treatment

time (hr)	CT(%)	100 red	35 blue	35 purple	50 pink	50 black	50 print	50 knit
-1(control)		27.88	42.98	42.84	44.76	89.06	37.99	33.22
		0.8	1.9	2.6	4.0	7.1	0.2	5.7
0(after hydrolysis)		57.49	5.49	4.28	12.76	14.51	10.90	63.98
		2.3	0.5	0.3	0.3	1.6	2.0	10.5
0.5		40.96	4.90	4.38	9.68	10.55	10.03	48.67
		4.7	0.4	0.3	0.3	1.2	1.3	6.8
1		31.21	4.12	4.07	8.95	6.59	8.34	30.88
		4.4	0.1	0.9	0.6	0.6	2.0	5.2
1.5		20.54	2.41	3.07	7.97	3.94	4.98	14.50
		1.5	0.2	0.6	0.0	0.3	0.8	0.5

VITA

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DOCTORAL EXAMINATION AND DISSERTATION REPORT

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Major Field: Human Ecology

Title of Dissertation: Complete Recycling of Textile Components from Cotton/Polyester Blended Fabrics

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